



1991

PERFORMANCE REPORT

DRINKING WATER ORGANICS

SECTION

MAY 1992

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Ministry
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Ministère
de
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Laboratory
Services
Branch

Direction des
services de
laboratoire

Mailing Address
125 Resources Road
P.O. Box 213
Rexdale, Ontario
M9W 5L1

Adresse postale
125, chemin resources
C.P. 213
Rexdale (Ontario)
M9W 5L1

July 8, 1992

MEMORANDUM

TO: Distribution List

FROM: S. Villard
DWO Section Manager

RE: DWO 1991 PERFORMANCE REPORT



Enclosed please find a copy of the 1991 Performance Report for the Drinking Water Organics Section. This report summarizes the actual performance of routine approved methods utilized in DWO Section laboratories in 1991 and provides information on quality control procedures adopted.

I would like to thank Bill Berg, Eva Duchoslav, David Hall, Eric Reiner and Vince Taguchi for the effort they put into realizing this report.

S. Villard

Enclosure
SV:ed:mb

DISTRIBUTION LIST

Gerry C. Ronan
Assistant Deputy Minister
Environmental Sciences & Standards
Division
135 St. Clair Ave. West, 14th Floor
Toronto, Ontario
M4V 1P5

Ed Piche
Director
Air Resources Branch
880 Bay St., 4th Floor
Toronto, Ontario
M5S 1Z8

David Cook
Manager, Program Coordination
Air Resources Branch
880 Bay St., 4th Floor
Toronto, Ontario
M5S 1Z8

Doug Harper
Manager, CAP/VES Implementation Team
Air Resources Branch
880 Bay St., 4th Floor
Toronto, Ontario
M5S 1Z8

Prasanta K. Misra
Manager, Air Quality & Meteorology
Air Resources Branch
125 Resources Road
Rexdale, Ontario
M4V 1K6

Maris Lusia
Manager, Atm. Research & Special Programs
Air Resources Branch
880 Bay St., 4th Floor
Toronto, Ontario
M5S 1Z8

Janine Zend
Manager, Emission Technology & Reg.
Development
Air Resources Branch
880 Bay St., 4th Floor
Toronto, Ontario
M5S 1Z8

Ron Pearson
Manager, Phytotoxicology
Air Resources Branch
880 Bay St., 4th Floor
Toronto, Ontario
M5S 1Z8

Ivy Wile
Director
Hazardous Contaminants Branch
2 St. Clair Ave. West, 12th Floor
Toronto, Ontario
M4V 1L5

John Onderdonk
Manager, Pesticides Section
Hazardous Contaminants Branch
2 St. Clair Ave. West, 12th Floor
Toronto, Ontario
M4V 1L5

Smith Jim
Assistant Director
Hazardous Contaminants Branch
2 St. Clair Ave. West, 12th Floor
Toronto, Ontario
M4V 1L5

Pat Hollett
Director
Investigations & Enforcement Branch
250 Davisville Ave., 5th Floor
Toronto, Ontario
M4S 1H2

Helle Tosine
Director
Research & Technology Branch
135 St. Clair Ave. West, 12th Floor
Toronto, Ontario
M4V 1P5

Jim Ashman
Director
Water Resources Branch
1 St. Clair Ave. West, 4th Floor
Toronto, Ontario
M4V 1K6

Gerry Rees
Assistant Director
Water Resources Branch
1 St. Clair Ave. West, 4th Floor
Toronto, Ontario
M4V 1K6

Ken Roberts
Manager, Drinking Water Section
Water Resources Branch
1 St. Clair Ave. West, 3rd Floor
Toronto, Ontario
M4V 1K6

Fred C. Fleischer
Manager, Great Lakes Section
Water Resources Branch
1 St. Clair Ave. West, 6th Floor
Toronto, Ontario
M4V 1K6

Peter Dillon
Manager, Limnology Section
Water Resources Branch
Bellwood Acres Road
Dorset, Ontario
P0A 1E0

Edward W. Turner
Manager, MISA / Industrial Section
Water Resources Branch
1 St. Clair Ave. West, 5th Floor
Toronto, Ontario
M4V 1K6

Steve E. Salbach
Senior Manager, MISA
Water Resources Branch
1 St. Clair Ave. West, 5th Floor
Toronto, Ontario
M4V 1K6

John D. Kinhead
Manager, Watershed Management Section
Water Resources Branch
1 St. Clair Ave. West, 7th Floor
Toronto, Ontario
M4V 1K6

Jim Merritt
Director
Central Region
Toronto Regional Office
7 Overlea Blvd, 4th Floor
Toronto, Ontario
M4H 1A8

Jan Beaver
Chief, Environmental Quality Assessment
Central Region
7 Overlea Blvd, 4th Floor
Toronto, Ontario
M4H 1A8

Ron Hore
Director
Northeastern Region
Sudbury Regional Office
199 Larch St., 11th Floor
Sudbury, Ontario
P3E 5P9

Raymond R. Potvin
Chief, Air Quality Assessment
Northeastern Region
199 Larch St., 11th Floor
Sudbury, Ontario
P3E 5P9

Jim Fry
Manager, Technical Assessment
Northeastern Region
199 Larch St., 11th Floor
Sudbury, Ontario
P3E 5P9

Wayne G. Scott
Director
Northwestern Region
Thunder Bay Regional Office
P.O. Box 5000
435 James St. South, 3rd Floor
Thunder Bay, Ontario
P7C 5G6

James D. Stasiuk
Manager, Thunger Bay Laboratory
Northwestern Region
421 James St. South
Thunder Bay, Ontario
P7E 2V6

Bill Creighton
Manager, Technical Assessment
Northwestern Region
P.O. Box 5000
435 James St. South, 3rd Floor
Thunder Bay, Ontario
P7C 5G6

Brian Ward
Director
Southeastern Region
Kingston Regional Office
133 Dalton Ave.
Kingston, Ontario
K7K 6C2

Paul Elliot
Chief, Air Quality Assessment
Southeastern Region
133 Dalton Ave.
Kingston, Ontario
K7K 6C2

Stan MacBeth
Chief, Kingston Laboratory
Southeastern Region
133 Dalton Ave.
Kingston, Ontario
K7K 6C2

Les Fitz
Manager, Technical Assessment
Southeastern Region
133 Dalton Ave.
Kingston, Ontario
K7K 6C2

D. A. McTavish
Director
Southwestern Region
985 Adelaide St. South
London, Ontario
N6E 1V3

W.G. Cook
Chief, Laboratory Services
Southwestern Region
985 Adelaide St. South
London, Ontario
N6E 1V3

J.R. Hatton
Manager, Technical Assessment
Southwestern Region
985 Adelaide St. South
London, Ontario
N6E 1V3

Hardy Wong
Director
West Central Region
119 King St. West
P.O. Box 2112, 12th Floor
Hamilton, Ontario
L8N 3Z9

D. Corr
Chief, Air Quality Assessment
West Central Region
36 Keefer Court
Unit 1
Hamilton, Ontario
L8E 4V1

Wayne Jackman
Chief, Water Resources Assessment
West Central Region
119 King St. West
P.O. Box 2112, 12th Floor
Hamilton, Ontario
L8N 3Z9

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HAZARDOUS CONTAMINANTS
COORDINATION BRANCH
135 ST. CLAIR AVENUE WEST
TORONTO, ONTARIO M4V 1P5

1991

PERFORMANCE REPORT
DRINKING WATER ORGANICS SECTION

Report Prepared By:

Eva Duchoslav (ed.)
Drinking Water Organics Section
Laboratory Services Branch
Ontario Ministry of the Environment

FEBRUARY 1992



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PIBS 1933E

The editor wishes to thank all Drinking Water Organics Section staff who helped to realize this report by providing experimental data and valuable comments.

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INTRODUCTION

This document represents the first publication of an annual summary of the performance of routine approved analytical methods within the laboratories of Drinking Water Organics Section.

The Drinking Water Organics Section is responsible for both qualitative and quantitative analyses of drinking, surface, river and lake waters for a wide array of organic chemicals, such as chlorinated benzenes, herbicides, pesticides, polynuclear aromatic hydrocarbons, extractable organics and purgeable organics at the part-per-trillion or part-per-billion level. In addition, analyses provided by the Section include ultra-trace quantitative analyses of environmental matrices for polychlorinated dibenzodioxins and polychlorinated dibenzofurans, most notably 2,3,7,8-tetrachlorodibenzo-p-dioxin. Also, DWO Section supports various Ministry's programs by providing characterization of unknown organic contaminants by mass spectrometry.

The major objective of the DWO Section's quality assurance program is to produce data of known quality, appropriate for a particular purpose. The quality control program is designed to detect any anomalies in the quality of the analytical results and to provide the basis for an immediate corrective action.

Within the DWO Section, the most common quality control tasks include the analyses of quality control samples, such as method blanks, fortified method blanks, samples fortified with surrogates, check calibration solutions and reference materials, and the interpretation of the resulting data. For each analytical method, the actual quality control procedures are described in detail in section 6.2 of the corresponding official method text.

This Performance Summary Report is based on the results of selected quality control samples acquired between January and December, 1991. In this report, each abstract of the analytical method is accompanied by corresponding performance charts and summary tables. Performance charts contained indicate the mean and the 99%-confidence limits for the variable presented.

METHOD CODE : OPTM-E3237A.1
METHOD TITLE: The Determination of Trihalomethanes in Water by Purge-and-Trap/Gas Chromatography

LABORATORY : Priority Pollutants Unit
SUPERVISOR : O.W. Berg

SAMPLE TYPE : surface water, groundwater, finished drinking water

PRINCIPLE OF THE METHOD :

Trihalomethanes are purged from an aqueous sample onto an adsorption trap, and subsequently, thermally desorbed onto a gas chromatographic capillary column. After separation, the organics are identified and quantified by Hall electrolytic conductivity detector.

PARAMETERS MEASURED :	LIS TEST CODE :	W (µg/L)	T (µg/L)
Chloroform	X1005J	0.5	5.0
Bromodichloromethane	X1010J	0.2	2.0
Dibromochloromethane	X1011J	0.2	2.0
Bromoform	X1015J	0.2	2.0
Total THM's	X2TTHM	0.5	5.0

REPORTING FORMAT :

Results are reported in parts per billion (µg/L) rounded off to the closest increment of W and up to maximum of three significant figures.

QUALITY CONTROL :

The routine quality control samples are designed to verify absence of potential contamination (method blanks) and to monitor validity of calibration (calibration solutions) and the agreement with the established method precision and accuracy (laboratory replicate samples, reference material).

The results for the analysis of calibration solution and the analysis of reference material have their control limits statistically derived.

REMARKS : In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

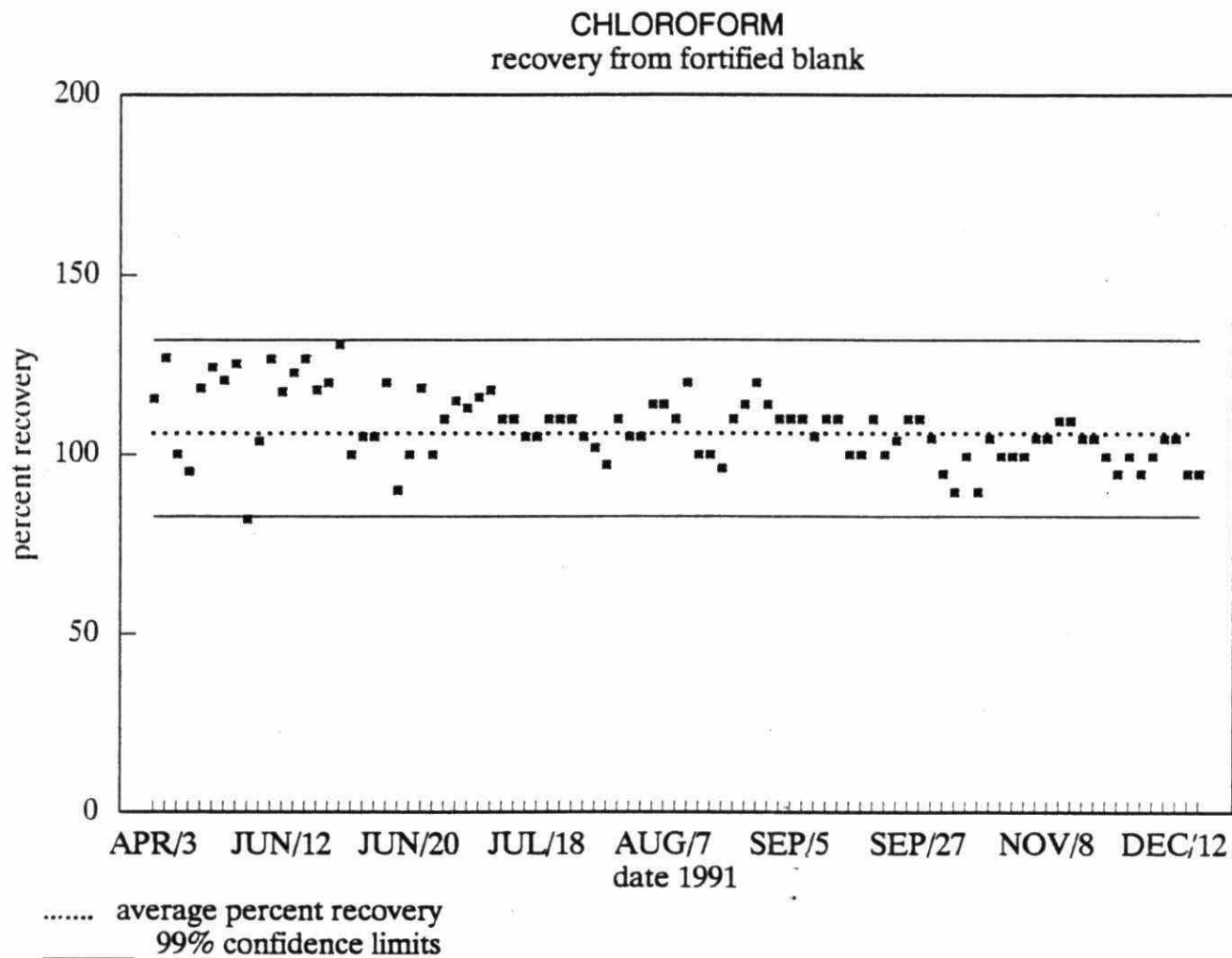
List of Performance Charts : Chloroform (recovery from fortified blank)
Bromodichloromethane (recovery from fortified blank)
Dibromochloromethane (recovery from fortified blank)
Bromoform (recovery from fortified blank)

List of Performance Tables : Method Blanks Summary
Chloroform
Bromodichloromethane
Dibromochloromethane
Bromoform

Method Blanks Summary

January 1991 - December 1991

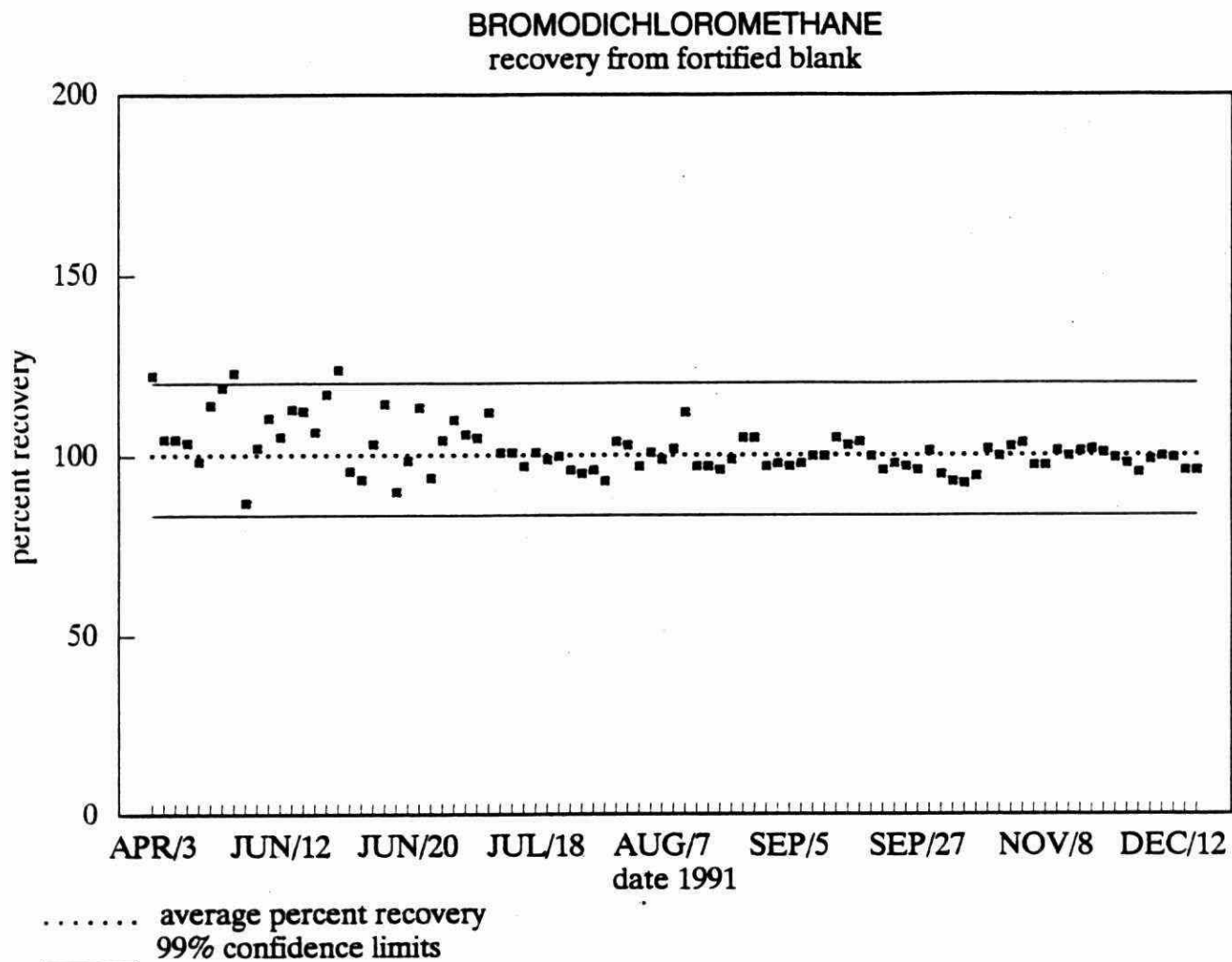
Analyte	Number of Observations	Average Concentration ($\mu\text{g/L}$)	Standard Deviation ($\mu\text{g/L}$)
chloroform	106	0.05	0.23
bromodichloromethane	106	0	0
dibromochloromethane	106	0	0
bromoform	106	0	0
THM's - total	106	0.05	0.23



Performance Summary Table

January - December 1991

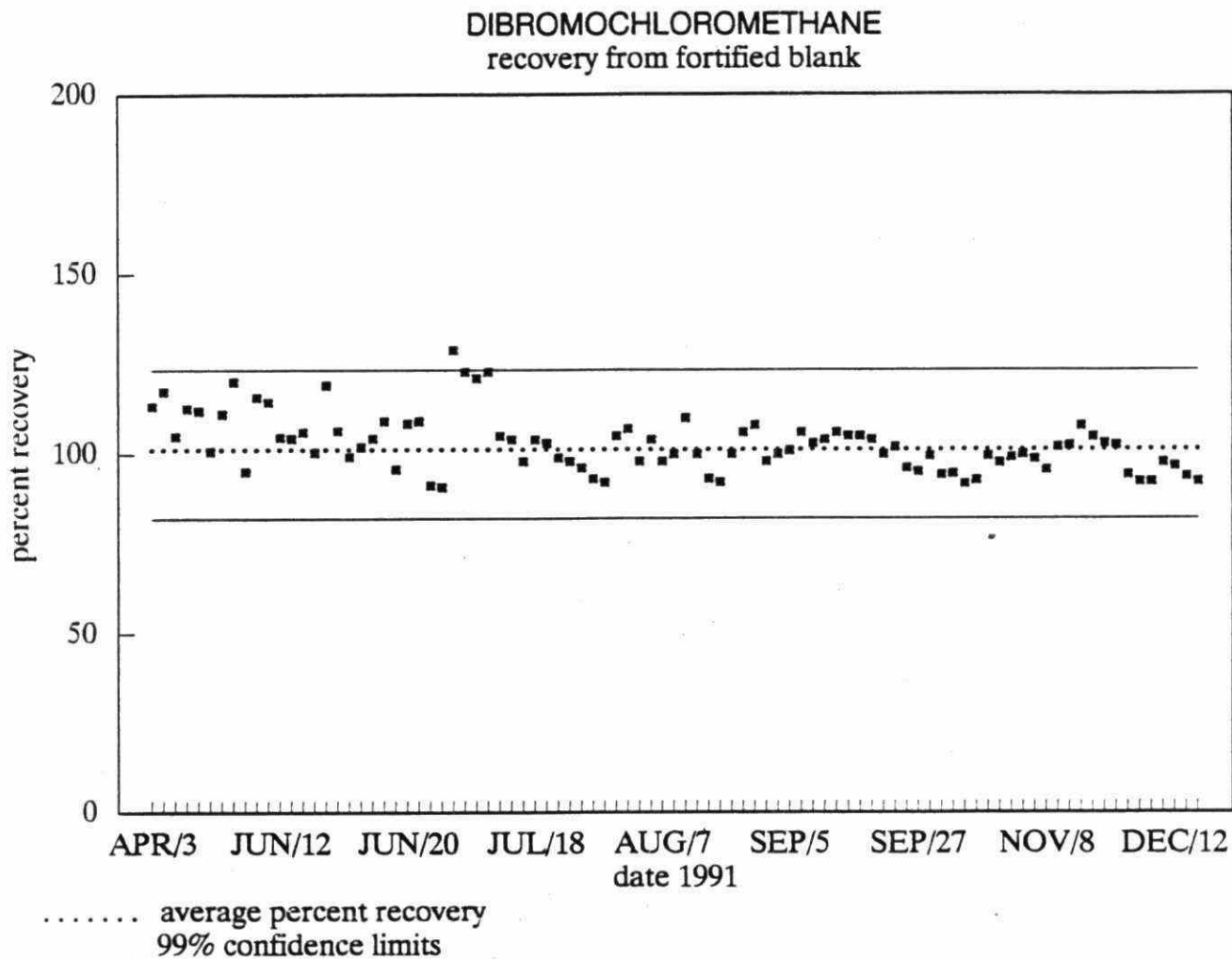
Analyte	chloroform
True Concentration	20 µg/L
Number of Observations	91
Within-run Rel. Standard Deviation	4% (n=13)
Between-run Standard Deviation	10%
Accuracy (% of expected)	107%



Performance Summary Table

January - December 1991

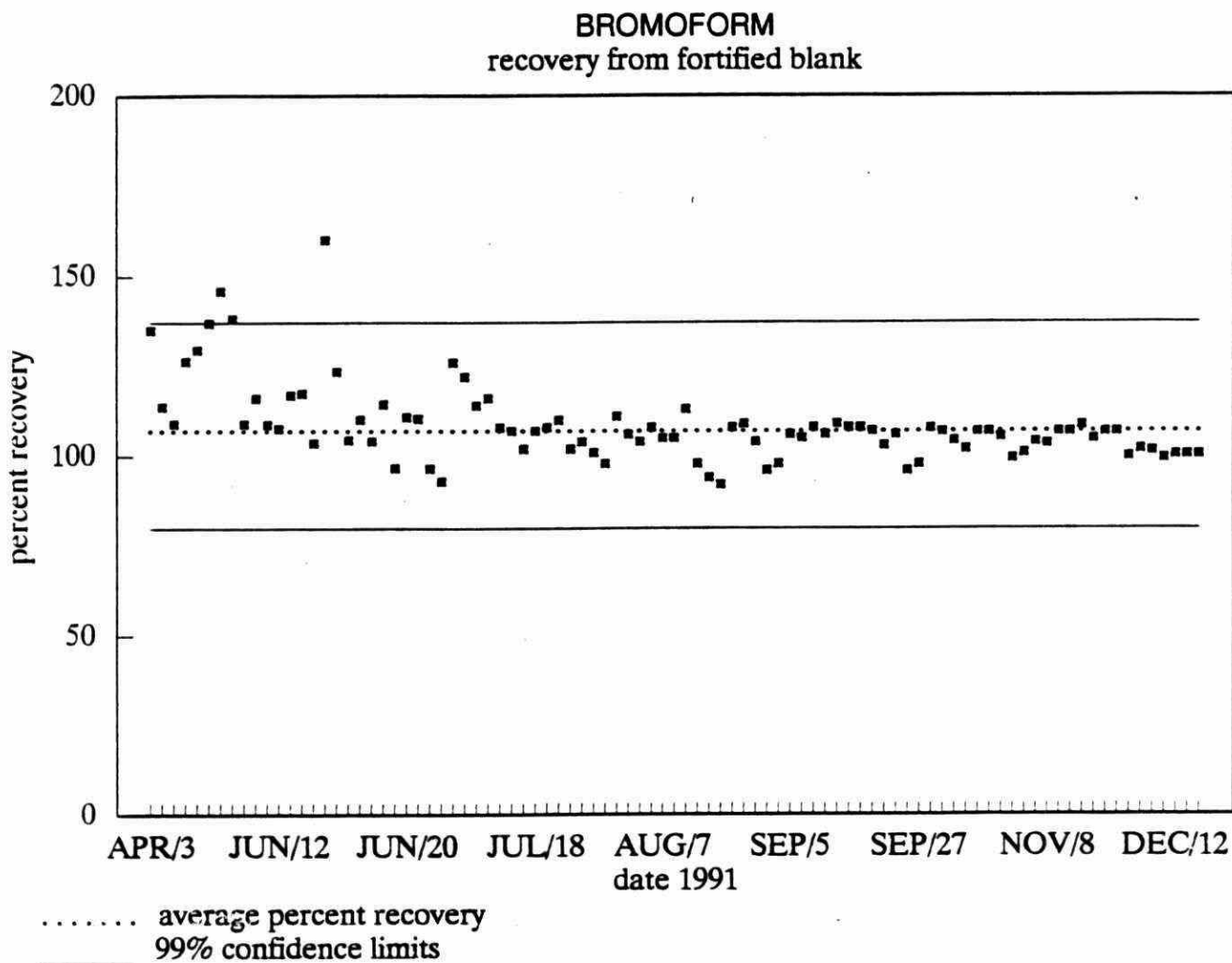
Analyte	bromodichloromethane
True Concentration	20 µg/L
Number of Observations	91
Within-run Rel. Standard Deviation	5% (n=13)
Between-run Standard Deviation	7%
Accuracy (% of expected)	102%



Performance Summary Table

January - December 1991

Analyte	dibromochloromethane
True Concentration	20 µg/L
Number of Observations	91
Within-run Rel. Standard Deviation	5% (n=13)
Between-run Standard Deviation	8%
Accuracy (% of expected)	103%



Performance Summary Table

January - December 1991

Analyte	bromoform
True Concentration	10 µg/L
Number of Observations	91
Within-run Rel. Standard Deviation	6% (n=13)
Between-run Standard Deviation	11%
Accuracy (% of expected)	108%

METHOD CODE : OPOV-E3144A.1
METHOD TITLE: The Determination of Purgeable Organic Compounds in Potable and Surface Waters by GC-FID and ECD
LABORATORY : Priority Pollutants Unit
SUPERVISOR : O.W. Berg
SAMPLE TYPE : surface water, groundwater, finished drinking water

PRINCIPLE OF THE METHOD :

This method involves purge-and-trap gas chromatographic analysis with simultaneous flame ionization and electron capture detection. The volatile organics compounds are purged from aqueous phase onto a Tenax trap by a stream of helium gas. The compounds are then thermally desorbed and cryofocussed onto one-metre piece of deactivated fused silica transfer line using liquid nitrogen as the coolant. The cold trap is then rapidly heated by an electric current and the compounds are swept into the chromatographic column.

The chromatogram resulting from the FID is used for quantification, while the ECD chromatogram is used mainly for confirmation.

PARAMETERS MEASURED :	LIS TEST CODE :	W (µg/L)	T (µg/L)
1,1-dichloroethene	X1001P	0.1	1.0
dichloromethane	X1002P	0.5	5.0
t-1,2-dichloroethene	X1003P	0.1	1.0
1,1-dichloroethane	X1004P	0.1	1.0
chloroform	X1005P	0.1	1.0
1,1,1-trichloroethane	X1006P	0.02	0.2
1,2-dichloroethane	X1007P	0.1	1.0
carbon tetrachloride	X1008P	0.2	2.0
benzene	B2001P	0.05	0.5
1,2-dichloropropane	X1009P	0.1	1.0
trichloroethylene	X1010P	0.1	1.0
bromodichloromethane	X1011P	0.1	1.0
toluene	B2002P	0.05	0.5
1,2-dibromoethane	X2EDB	0.1	1.0
1,1,2-trichloroethane	X1012P	0.1	1.0
dibromochloromethane	X1013P	0.1	1.0
tetrachloroethene	X1014P	0.05	0.5
chlorobenzene	X2001P	0.05	0.5
ethylbenzene	B2003P	0.05	0.5
m-xylene	B2005P	0.1	1.0
p-xylene	B2004P	0.1	1.0

(parameters measured continued)

bromoform	X1015P	0.2	2.0
styrene	B2008P	0.05	0.5
o-xylene	B2006P	0.05	0.5
1,1,2,2-tetrachloroethane	X1016	0.2	2.0
1,4-dichlorobenzene	X2002P	0.1	1.0
1,3-dichlorobenzene	X2003P	0.1	1.0
1,2-dichlorobenzene	X2004P	0.1	1.0
total trihalomethanes	X2TTHM	0.5	5.0

REPORTING FORMAT :

Results are reported in parts per billion ($\mu\text{g/L}$) rounded off to the closest increment of W and up to maximum of three significant figures.

QUALITY CONTROL :

The routine quality control operations monitor absence of potential interferences (method blanks) and consistency with the predetermined method performance (fortified method blanks).

REMARKS : This analytical method was modified in September 1991. The detection system was replaced with dual capillary gas chromatography with dual flame ionization detectors.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB QM Office.

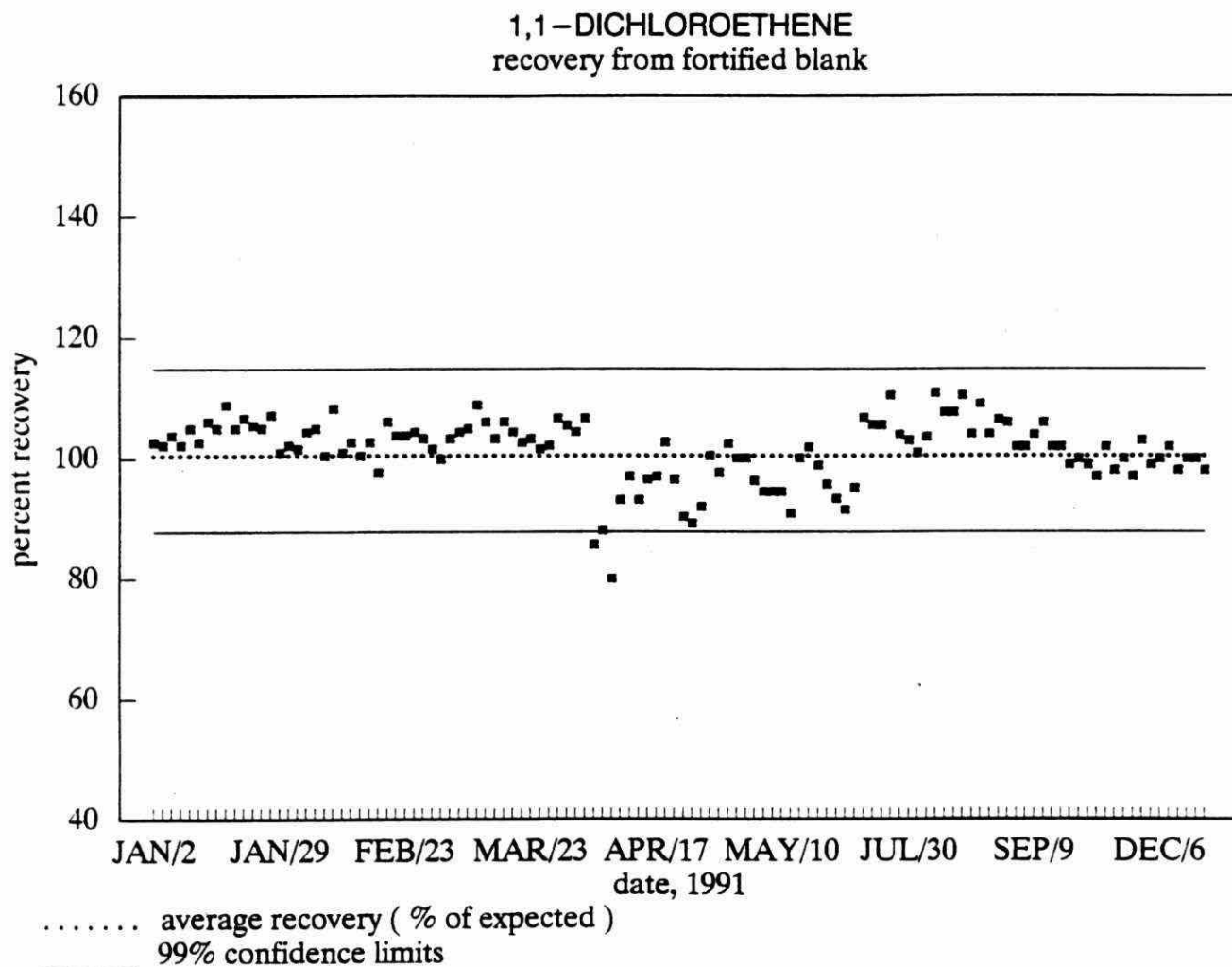
List of Performance Charts : all analytes described as parameters measured (recoveries from fortified blanks)

List of Performance Tables : Method Blanks Summary
all analytes described as parameters measured

Method Blanks Summary

January 1991 - December 1991

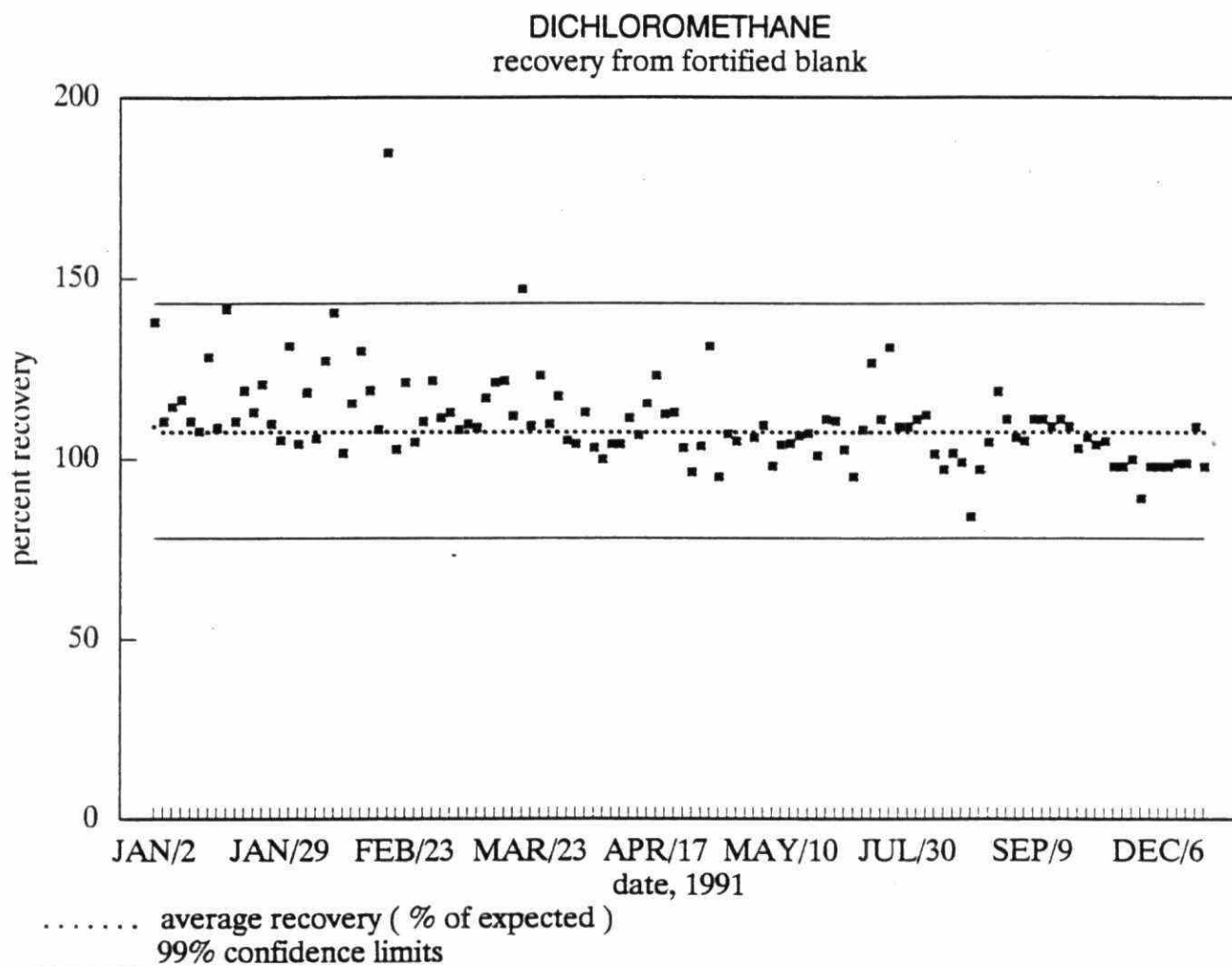
Analyte	Number of Observations	Average Concentration (µg/L)	Standard Deviation (µg/L)
1,1-dichloroethene	205	0	0
dichloromethane	205	0.40	0.93
t-1,2-dichloroethene	205	0	0
1,1-dichloroethane	205	0	0
chloroform	205	0.0007	0.0074
1,1,1-trichloroethane	205	0	0
1,2-dichloroethane	205	0	0
carbon tetrachloride	205	0	0
benzene	205	0.0038	0.0053
1,2-dichloropropane	205	0	0
trichloroethene	205	0.0004	0.0063
bromodichloromethane	205	0.0001	0.0010
toluene	205	0.0078	0.0075
1,2-dibromoethane	205	0	0
1,1,2-trichloroethane	205	0	0
dibromochloromethane	205	0	0
tetrachloroethene	205	0	0
chlorobenzene	205	0	0
ethylbenzene	205	0.0004	0.0025
m-xylene / p-xylene	205	0.0027	0.0050
bromoform	205	0	0
styrene	205	0.0027	0.0066
o-xylene	205	0.0008	0.0027
1,1,2,2-tetrachloroethane	205	0.001	0.018
1,4-dichlorobenzene	205	0.006	0.012
1,3-dichlorobenzene	205	0.004	0.011
1,2-dichlorobenzene	205	0.005	0.013
total trihalomethanes	205	0.0009	0.0074



Performance Summary Table

January - December 1991

Analyte	1,1-dichloroethene
True Concentration	1.77 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	2.2% (n=4)
Between-run Standard Deviation	5.2%
Accuracy (% of expected)	101%

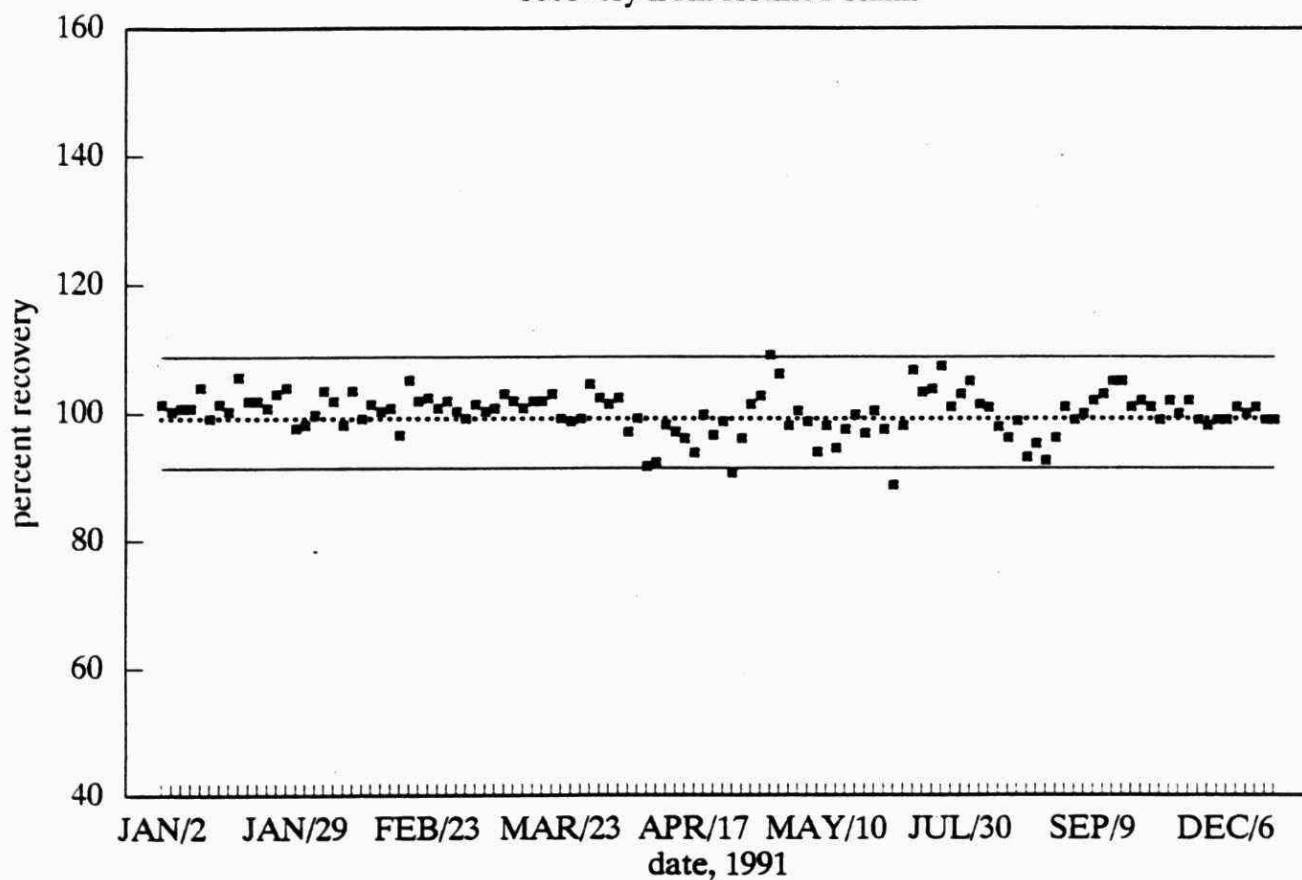


Performance Summary Table

January - December 1991

Analyte	dichloromethane
True Concentration	1.95 µg/L, 3.68 µg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1% (n=4)
Between-run Standard Deviation	12%
Accuracy (% of expected)	111%

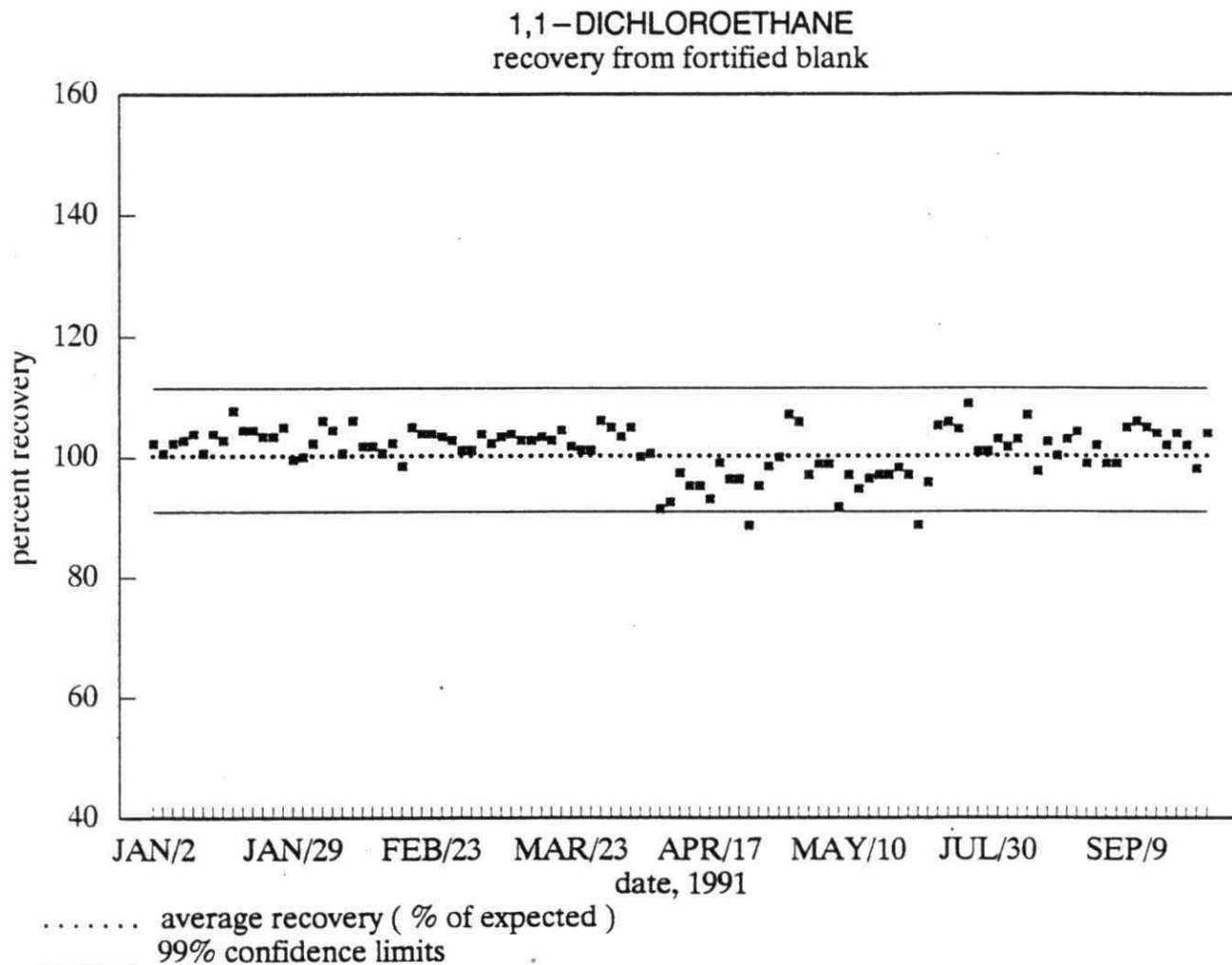
t-1,2-DICHLOROETHENE
recovery from fortified blank



Performance Summary Table

January - December 1991

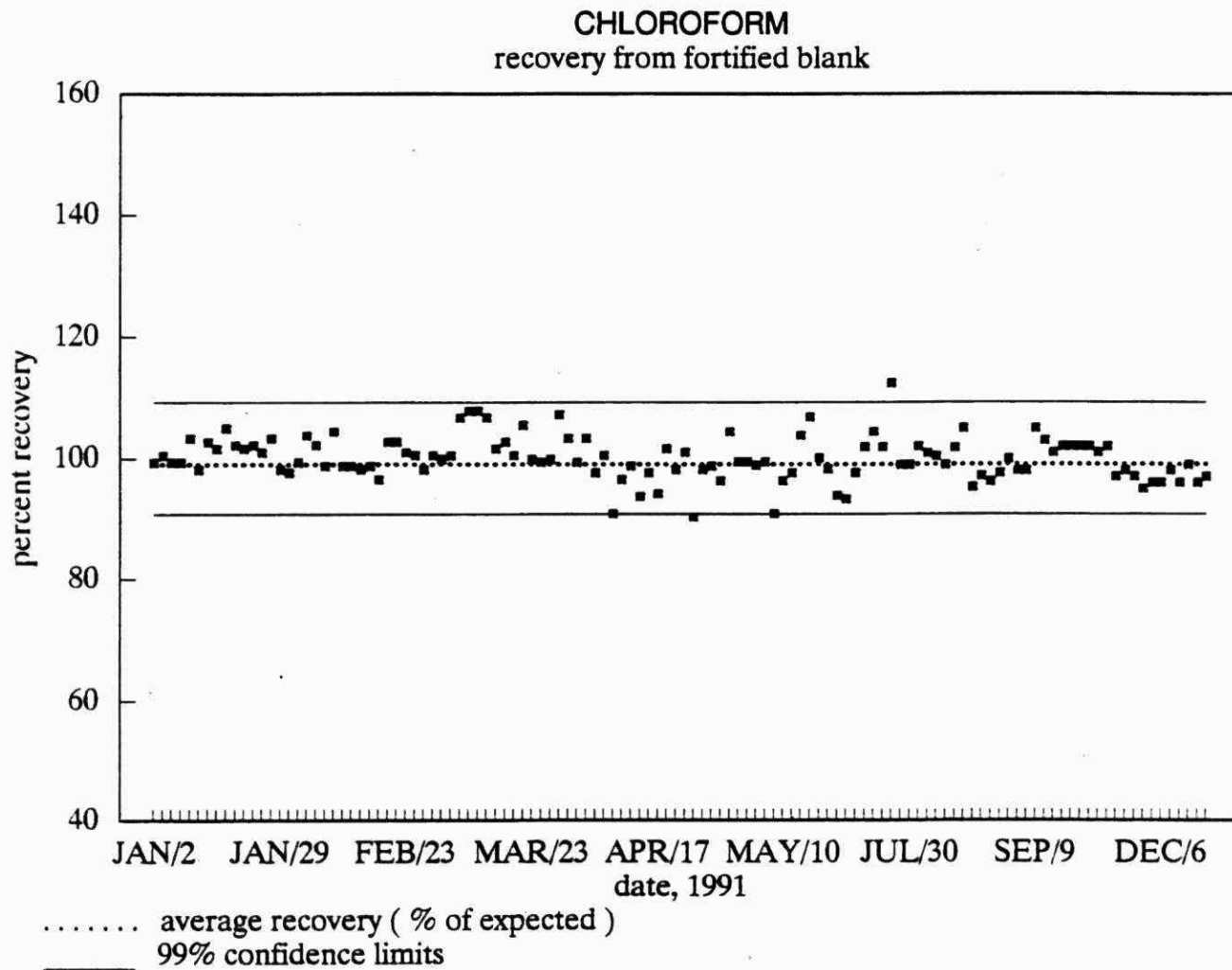
Analyte	t-1,2-dichloroethene
True Concentration	1.88 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.3% (n=4)
Between-run Standard Deviation	3.4%
Accuracy (% of expected)	100%



Performance Summary Table

January - December 1991

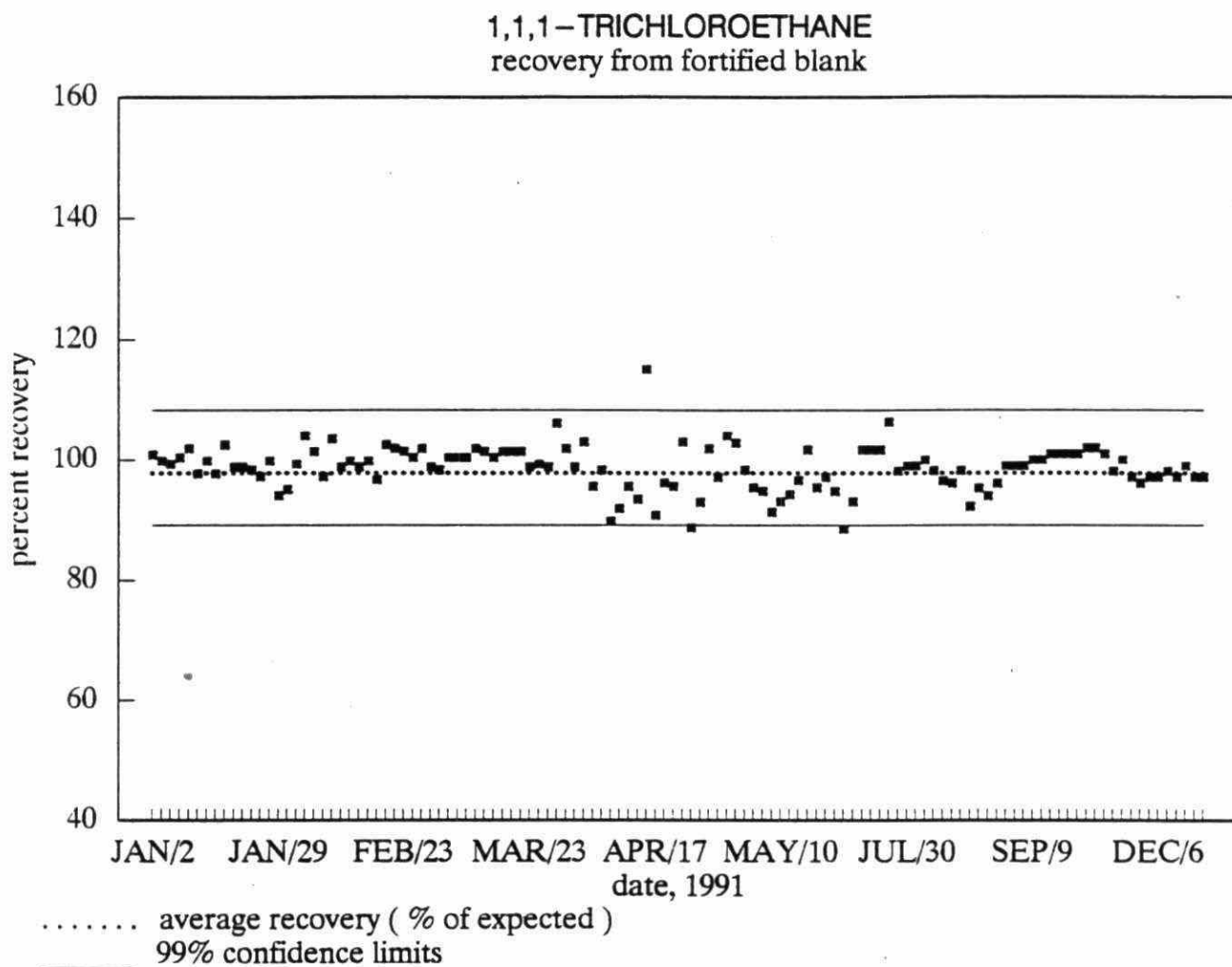
Analyte	1,1-dichloroethane
True Concentration	1.84 µg/L, 3.68 µg/L
Number of Observations	107
Within-run Rel. Standard Deviation	1.1% (n=4)
Between-run Standard Deviation	4.0%
Accuracy (% of expected)	101%



Performance Summary Table

January - December 1991

Analyte	chloroform
True Concentration	1.77 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% (n=4)
Between-run Standard Deviation	3.6%
Accuracy (% of expected)	100%

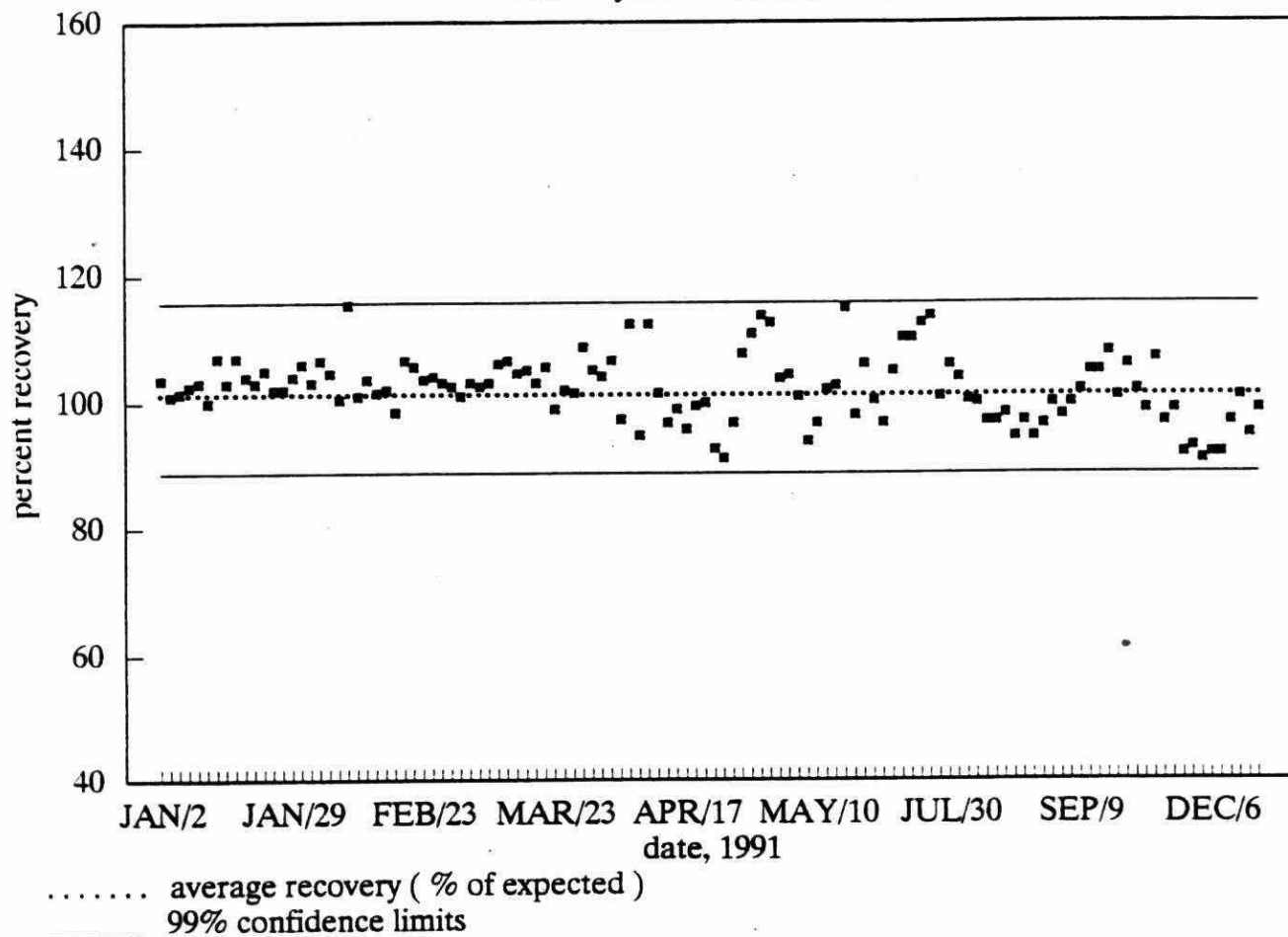


Performance Summary Table

January - December 1991

Analyte	1,1,1-trichloroethane
True Concentration	1.89 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% (n=4)
Between-run Standard Deviation	3.7%
Accuracy (% of expected)	98.6%

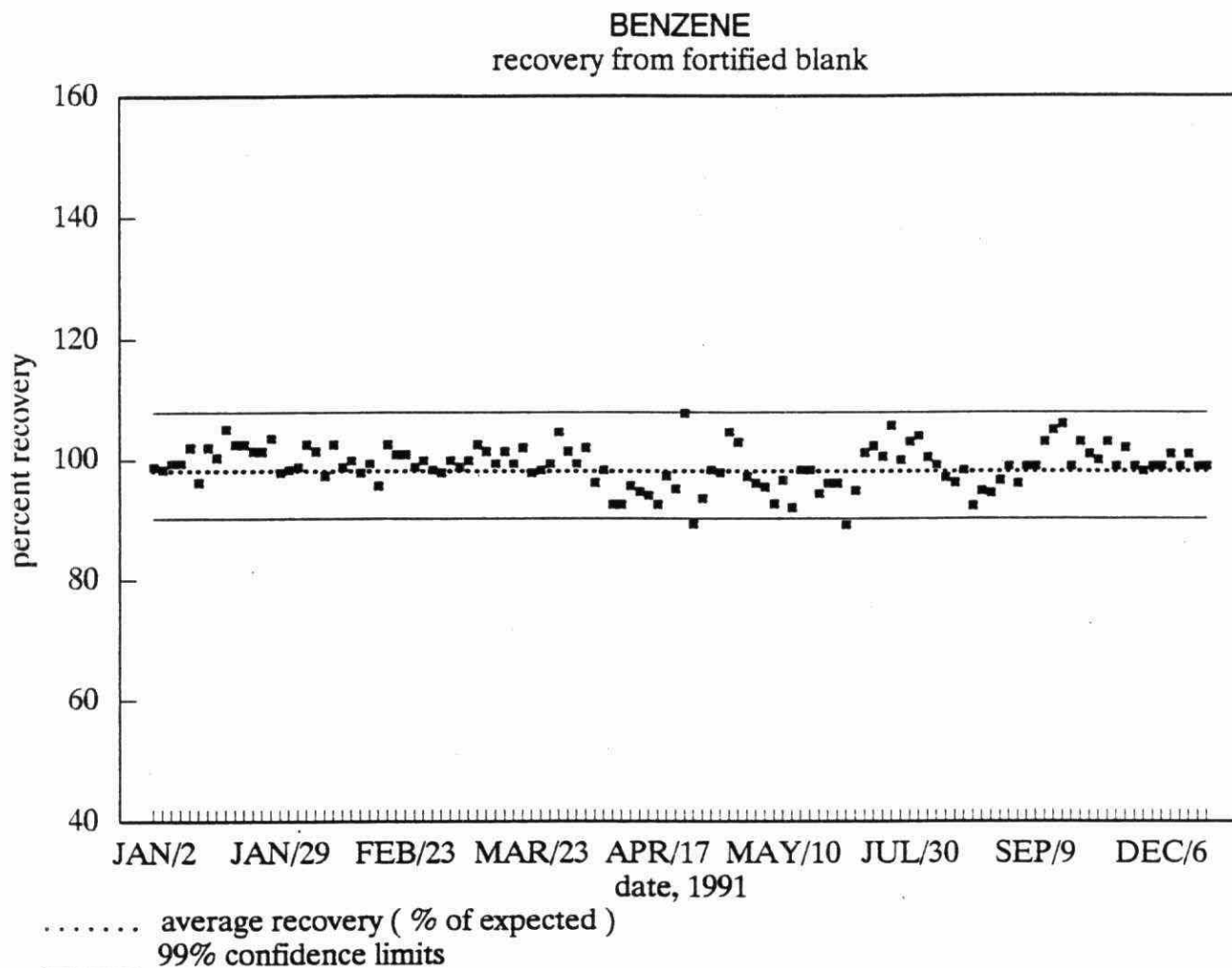
TETRACHLOROMETHANE
recovery from fortified blank



Performance Summary Table

January - December 1991

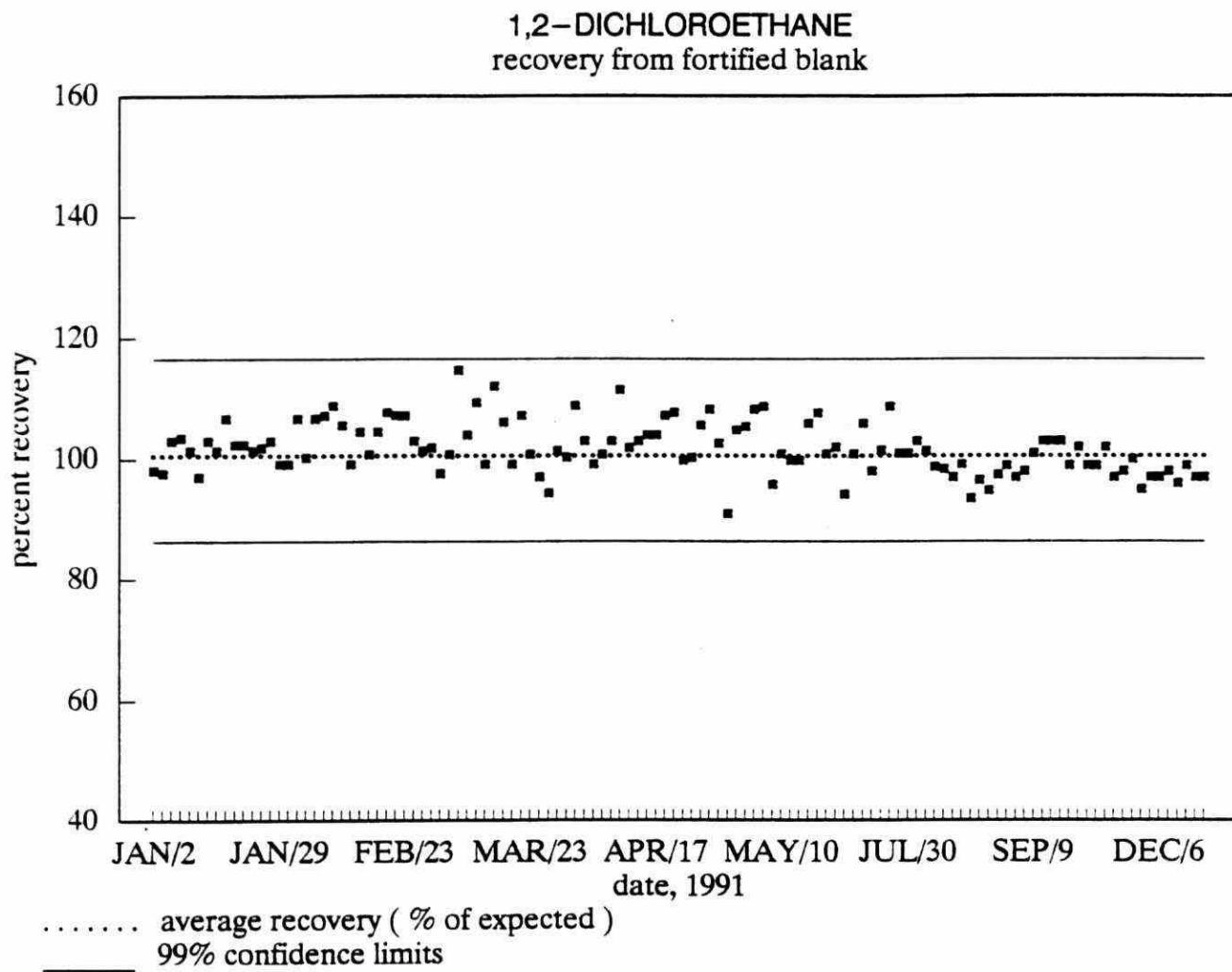
Analyte	tetrachloromethane
True Concentration	1.94 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.7% (n=4)
Between-run Standard Deviation	5.2%
Accuracy (% of expected)	102.2%



Performance Summary Table

January - December 1991

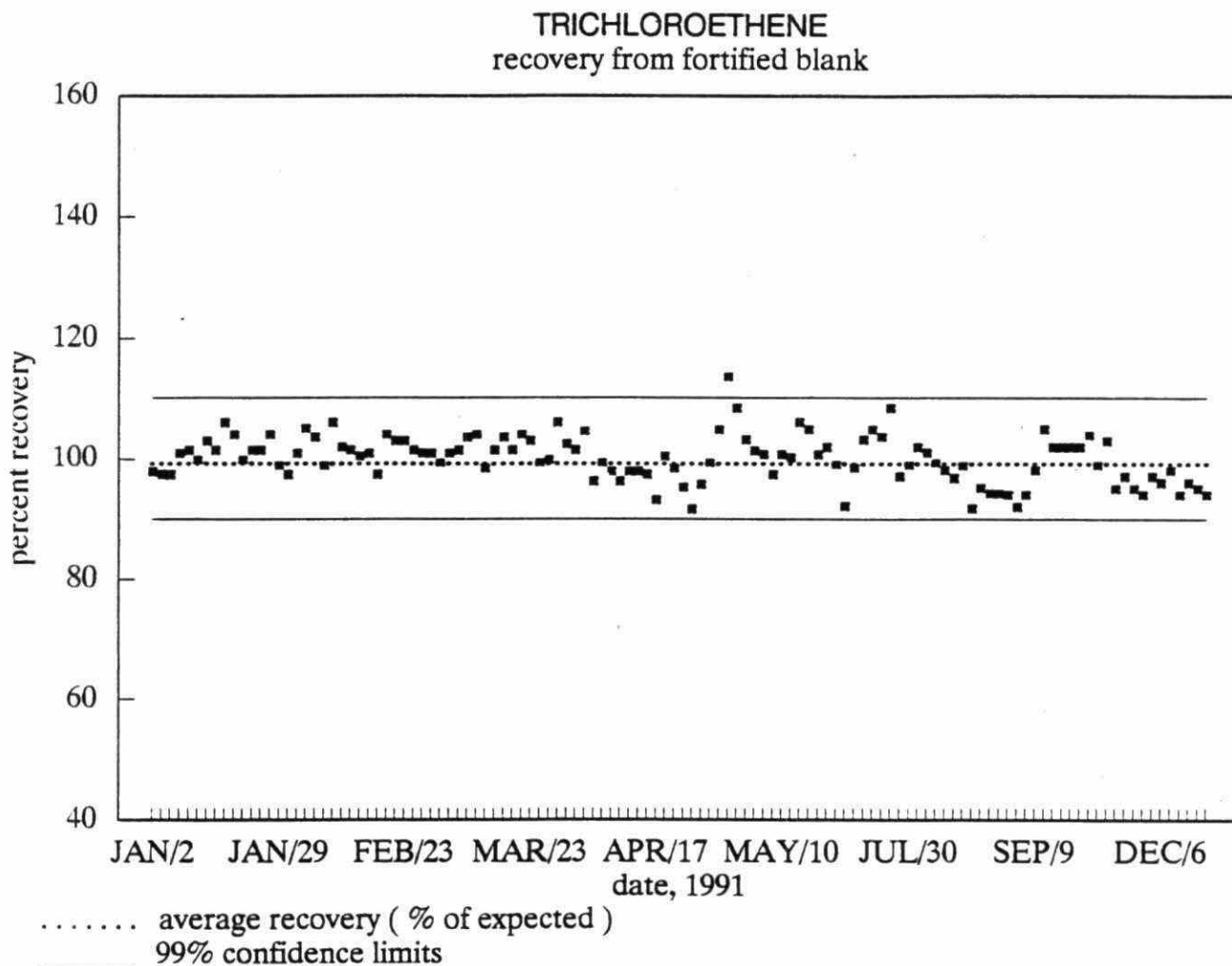
Analyte	benzene
True Concentration	1.90 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.3% (n=4)
Between-run Standard Deviation	3.4%
Accuracy (% of expected)	99%



Performance Summary Table

January - December 1991

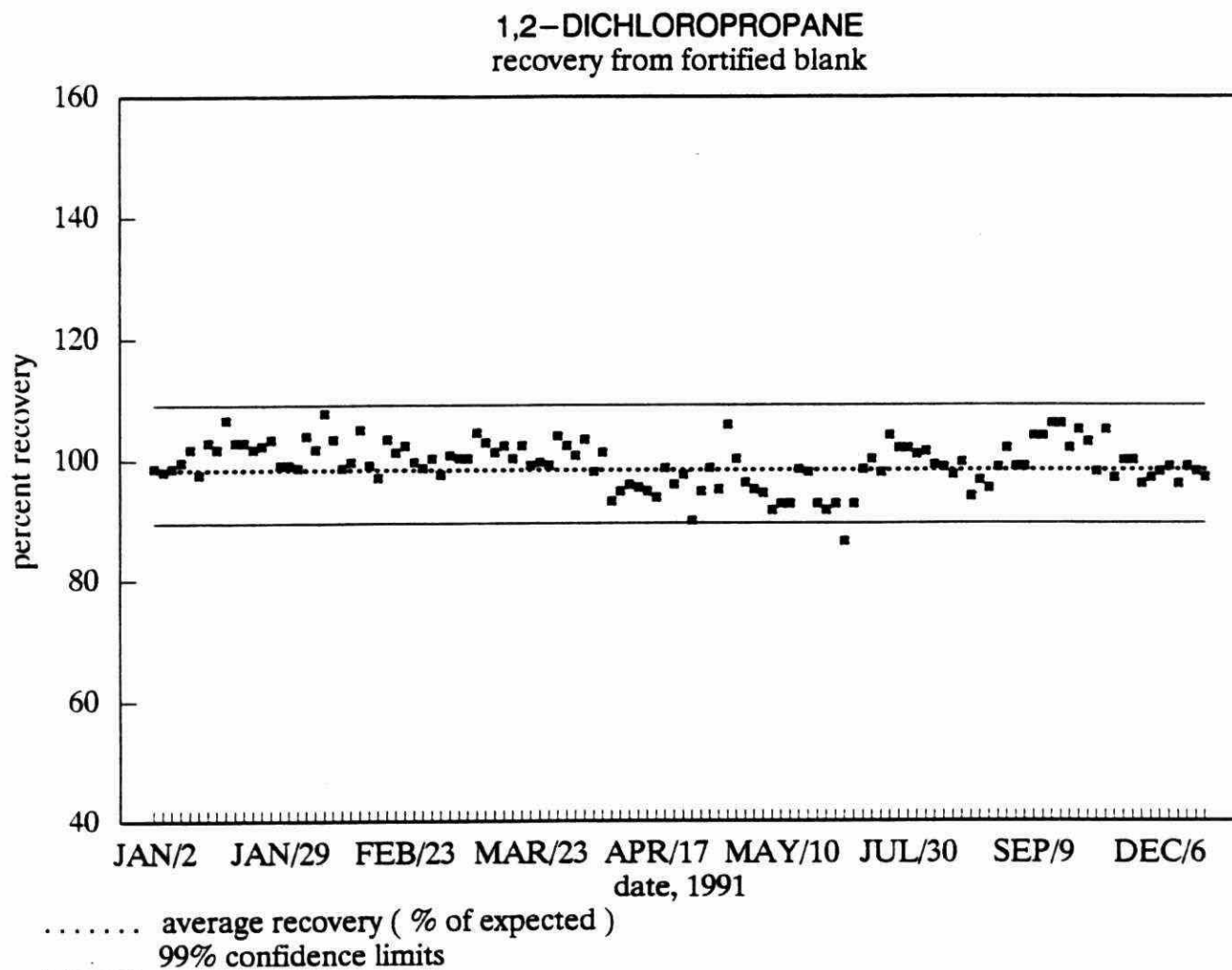
Analyte	1,2-dichloroethane
True Concentration	1.88 µg/L, 3.68 µg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1.0% (n=4)
Between-run Standard Deviation	5.8%
Accuracy (% of expected)	101.5%



Performance Summary Table

January - December 1991

Analyte	trichloroethene
True Concentration	1.92 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% (n=4)
Between-run Standard Deviation	3.9%
Accuracy (% of expected)	100%

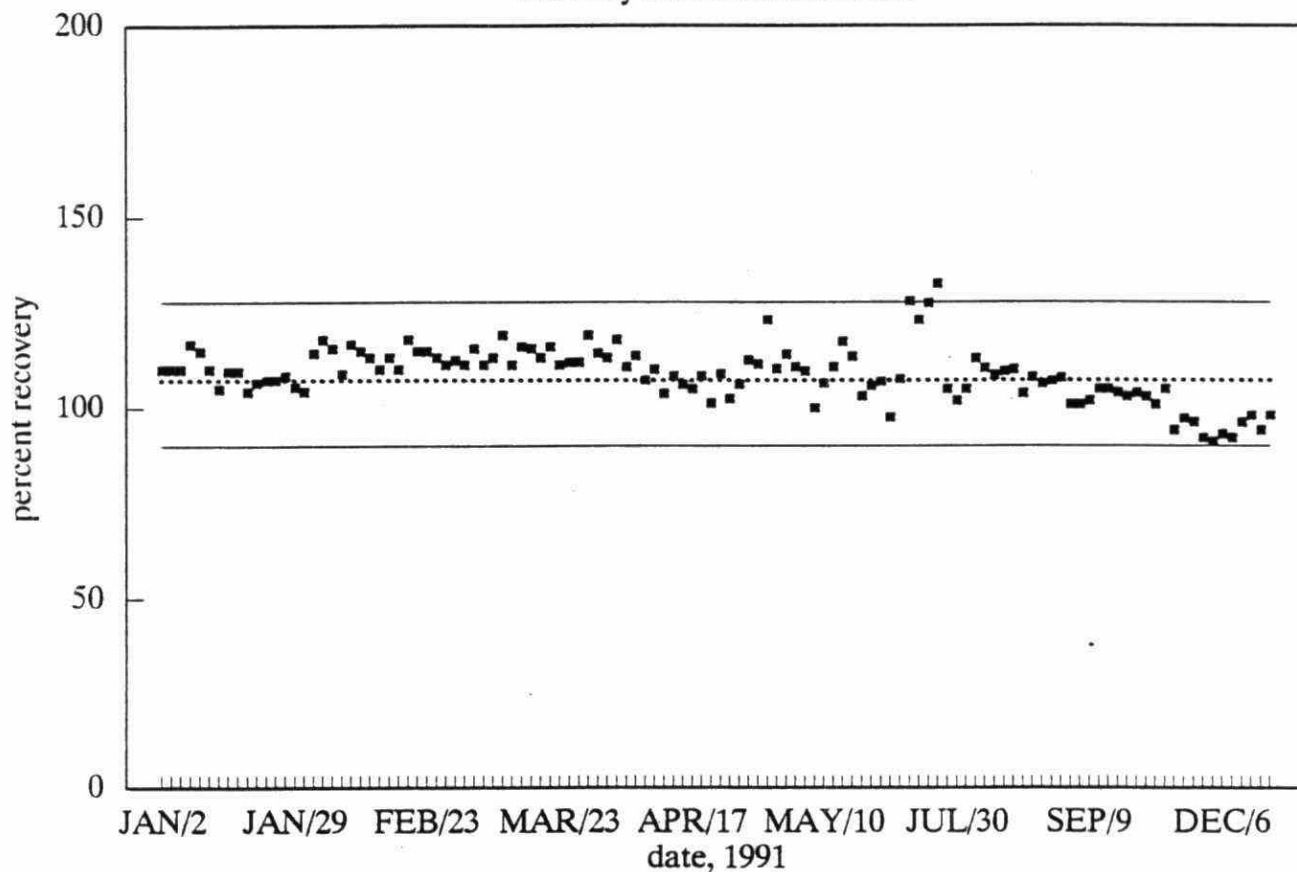


Performance Summary Table

January - December 1991

Analyte	1,2-dichloropropane
True Concentration	1.87 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	0.9% (n=4)
Between-run Standard Deviation	3.7%
Accuracy (% of expected)	99%

BROMODICHLOROMETHANE
recovery from fortified blank

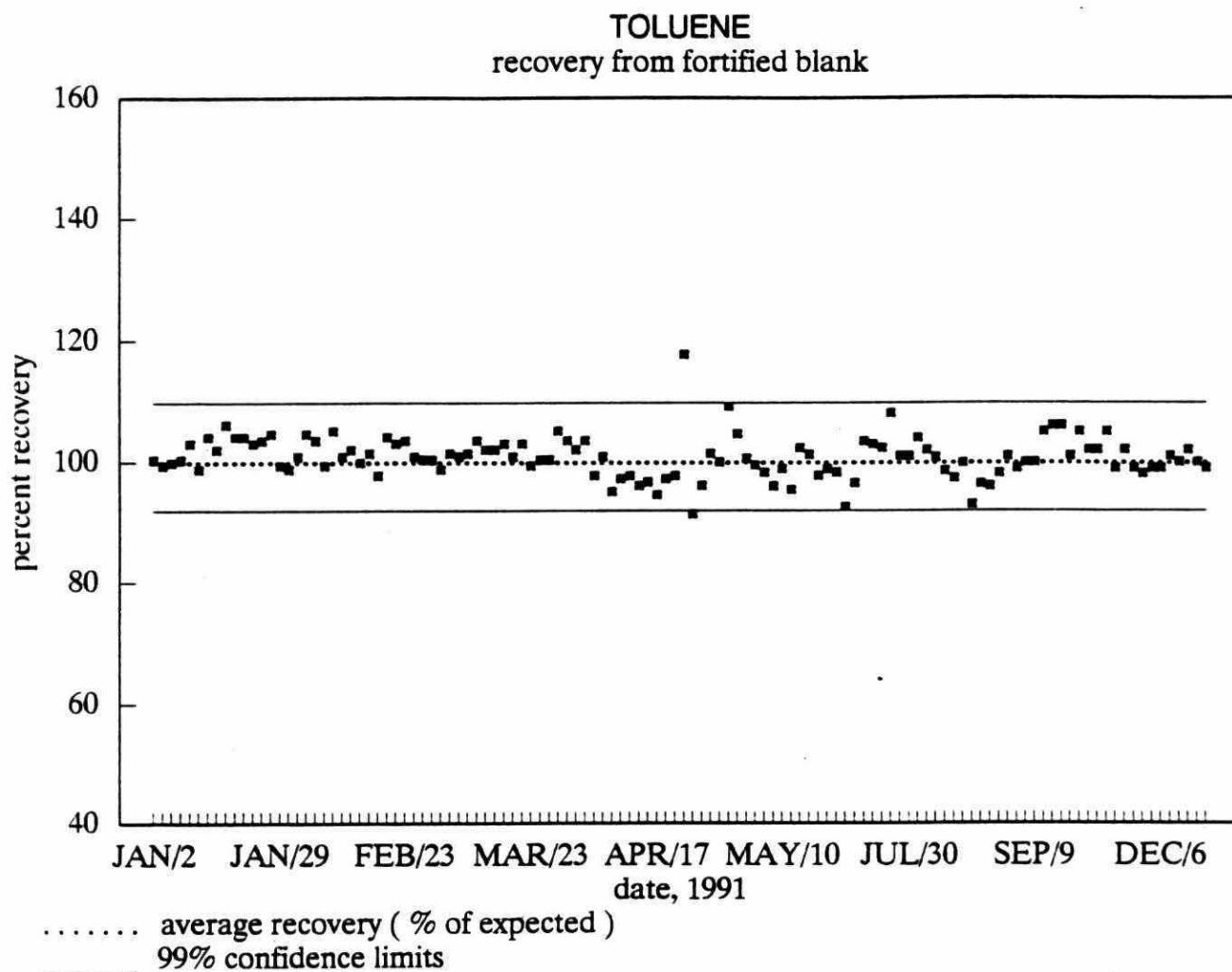


..... average recovery (% of expected)
—— 99% confidence limits

Performance Summary Table

January - December 1991

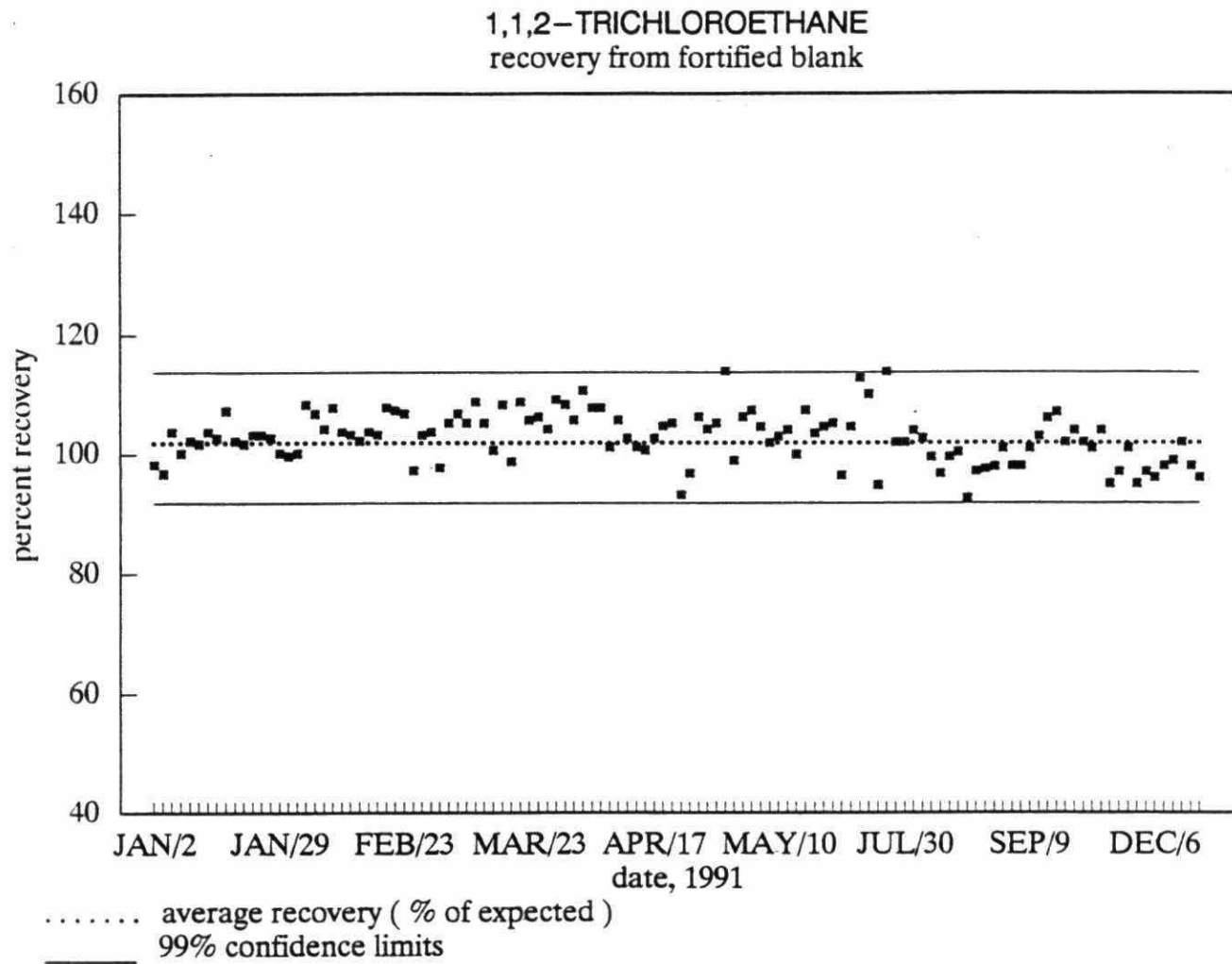
Analyte	bromodichloromethane
True Concentration	1.70 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% (n=4)
Between-run Standard Deviation	7.4%
Accuracy (% of expected)	110%



Performance Summary Table

January - December 1991

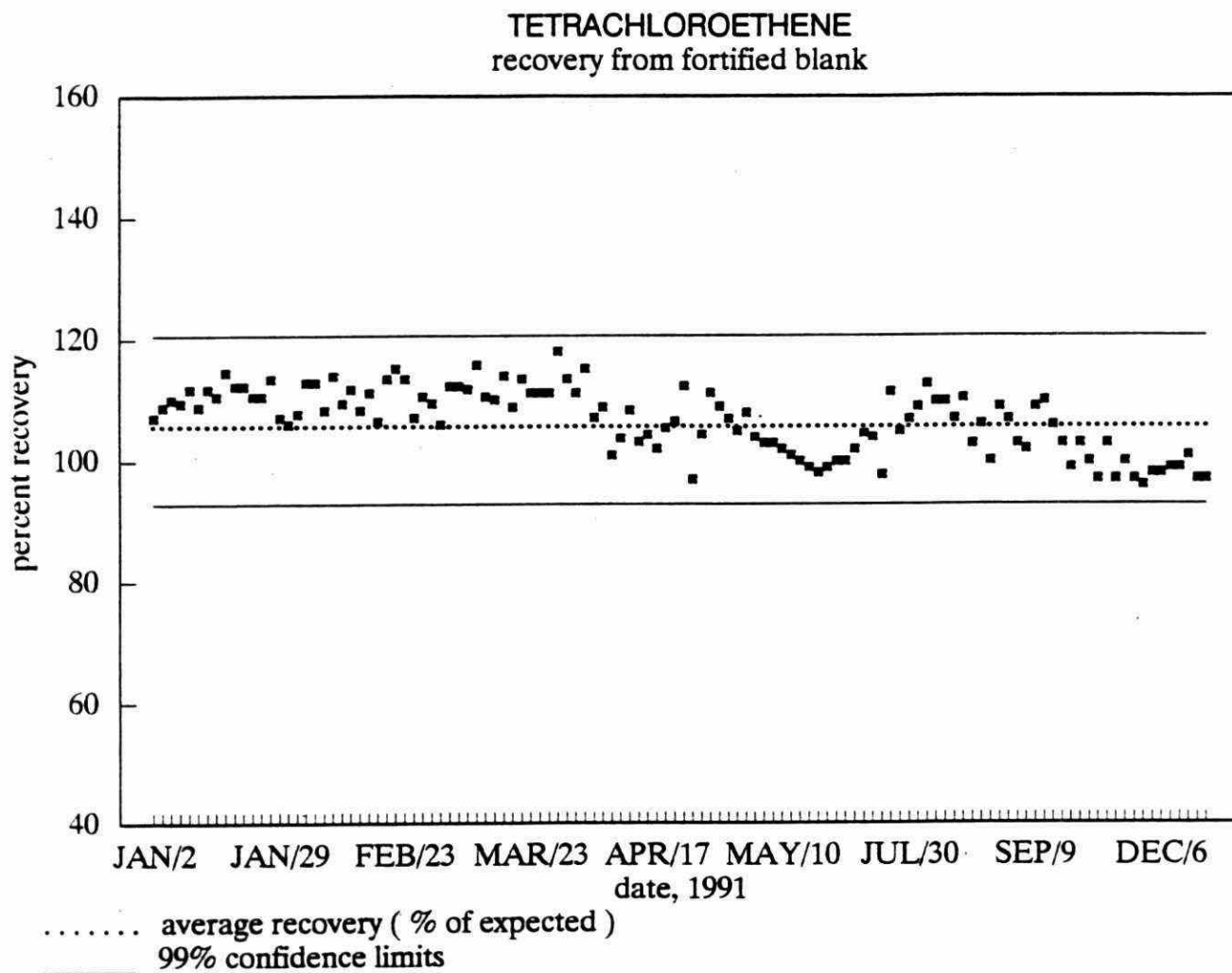
Analyte	toluene
True Concentration	1.90 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.5% (n=4)
Between-run Standard Deviation	3.5%
Accuracy (% of expected)	101%



Performance Summary Table

January - December 1991

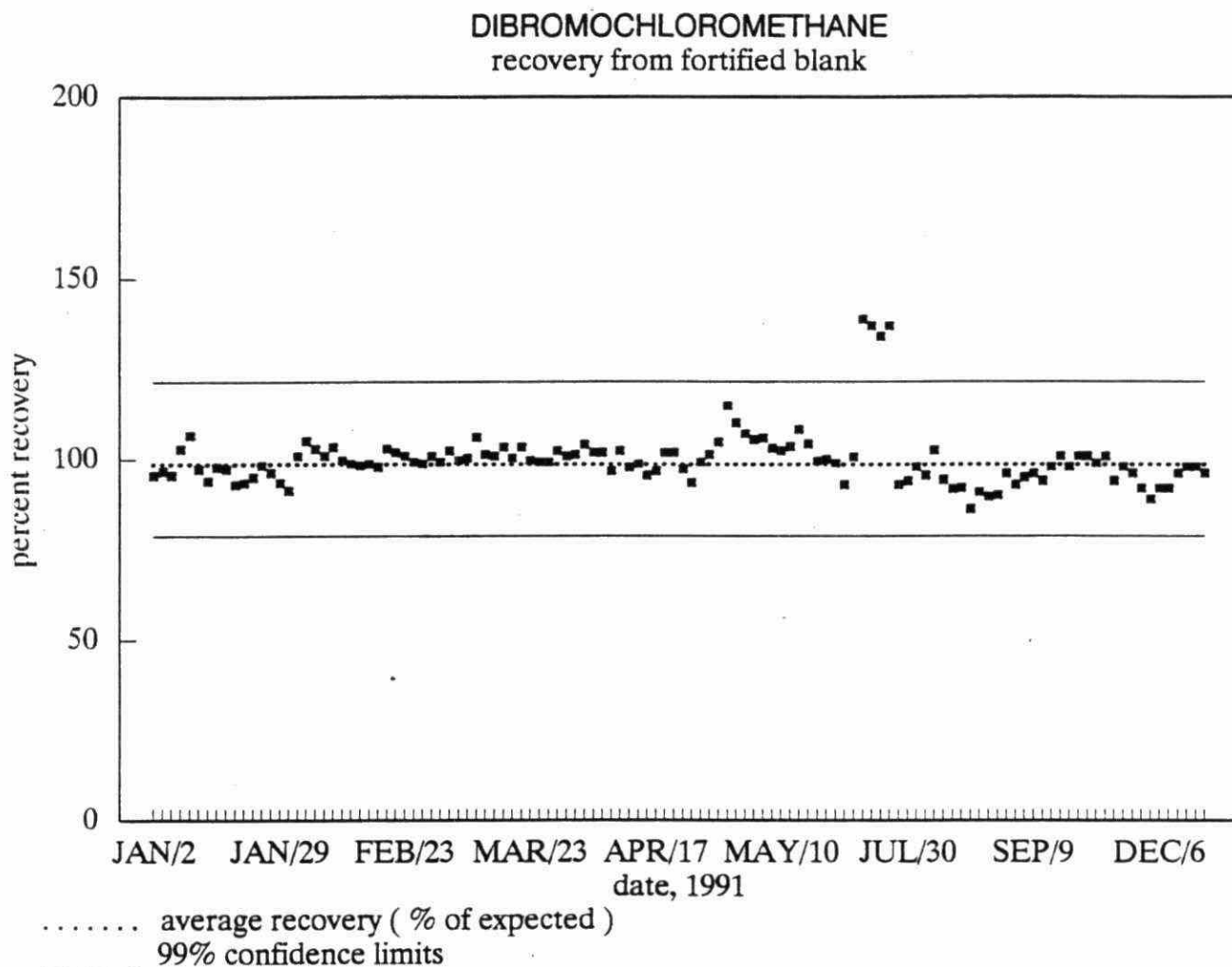
Analyte	1,1,2-trichloroethane
True Concentration	2.01 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% (n=4)
Between-run Standard Deviation	4.2%
Accuracy (% of expected)	103%



Performance Summary Table

January - December 1991

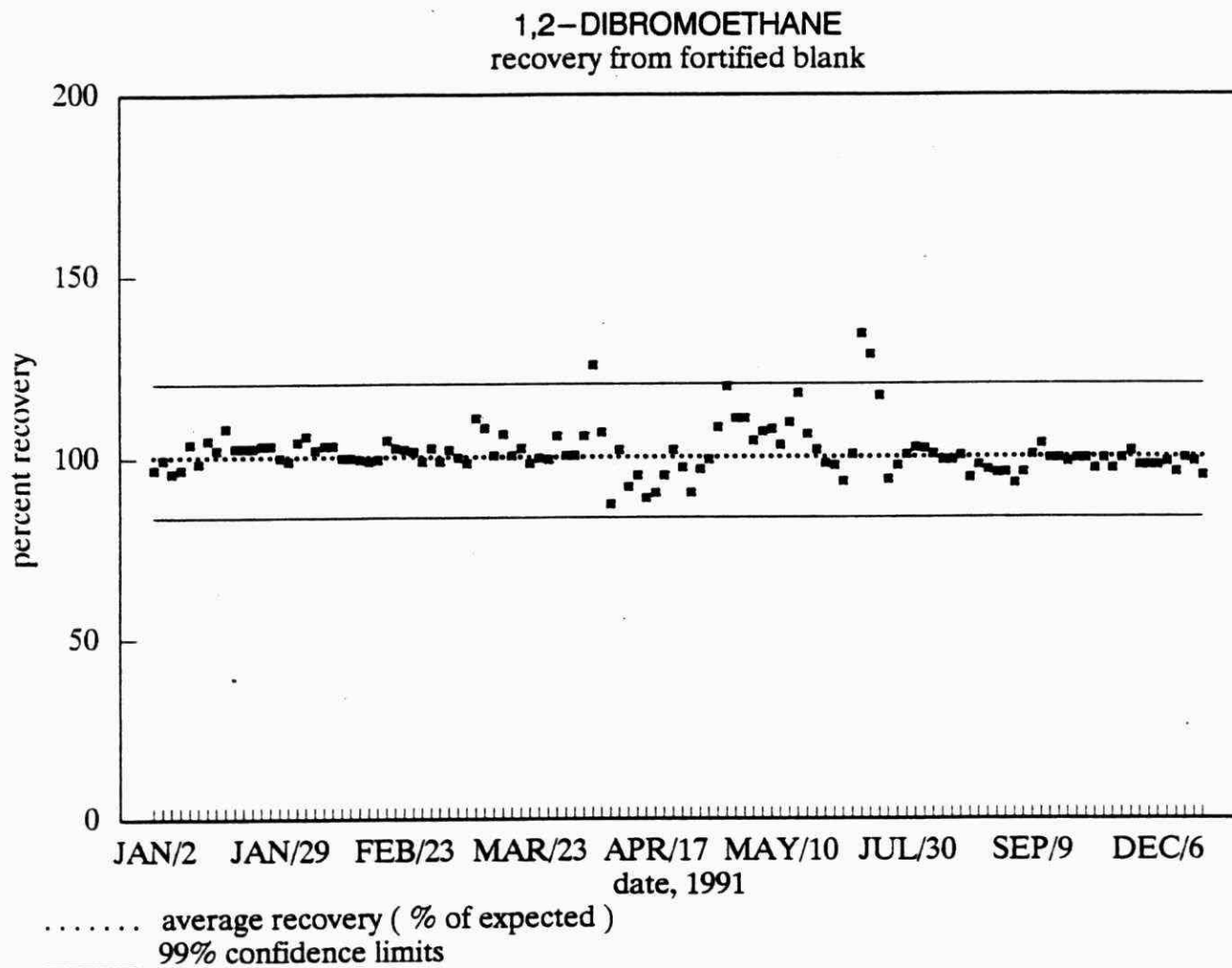
Analyte	tetrachloroethene
True Concentration	1.74 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.7% (n=4)
Between-run Standard Deviation	5.4%
Accuracy (% of expected)	107%



Performance Summary Table

January - December 1991

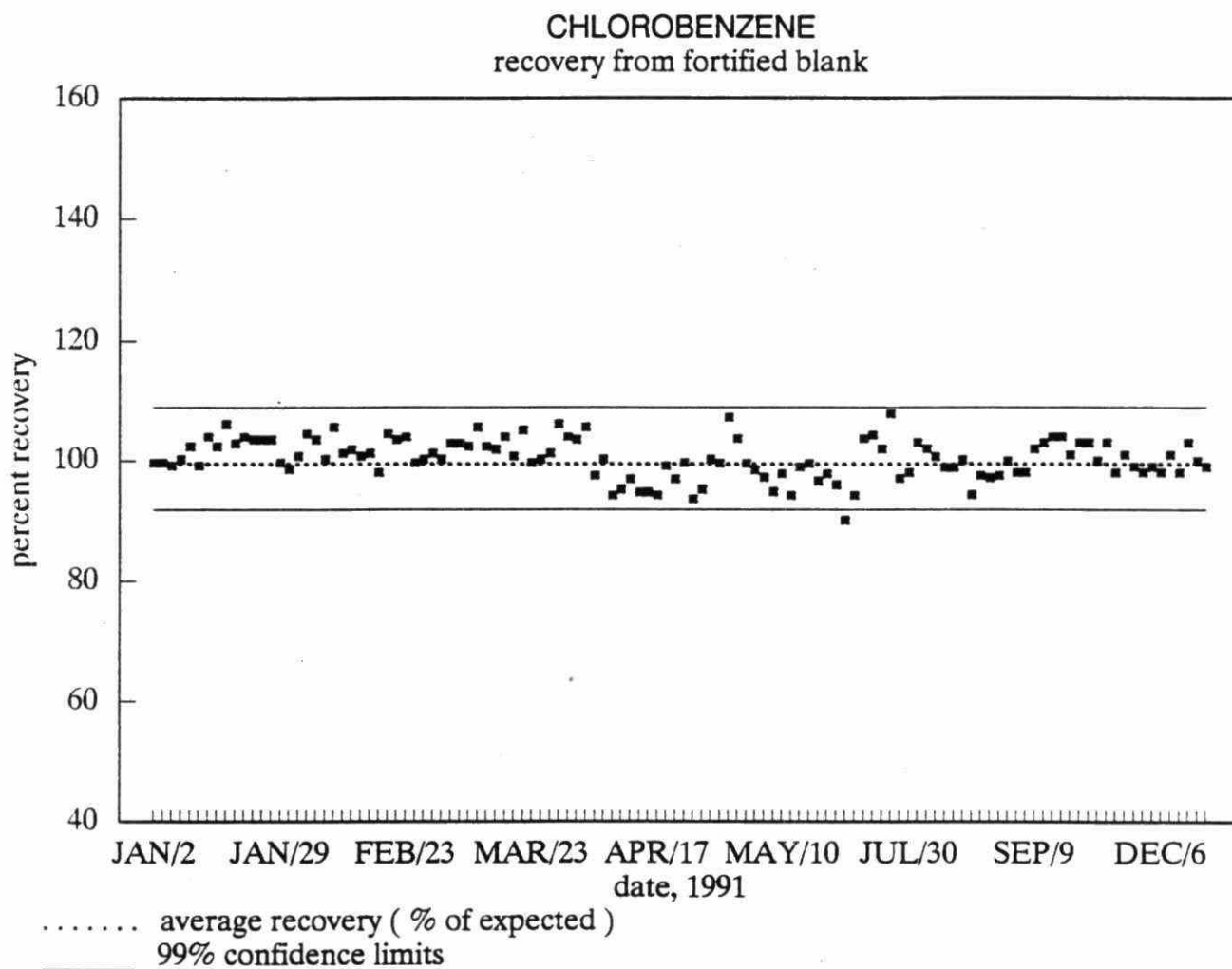
Analyte	dibromochloromethane
True Concentration	1.90 µg/L, 6.44 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.9% (n=4)
Between-run Standard Deviation	8.2%
Accuracy (% of expected)	100.1%



Performance Summary Table

January - December 1991

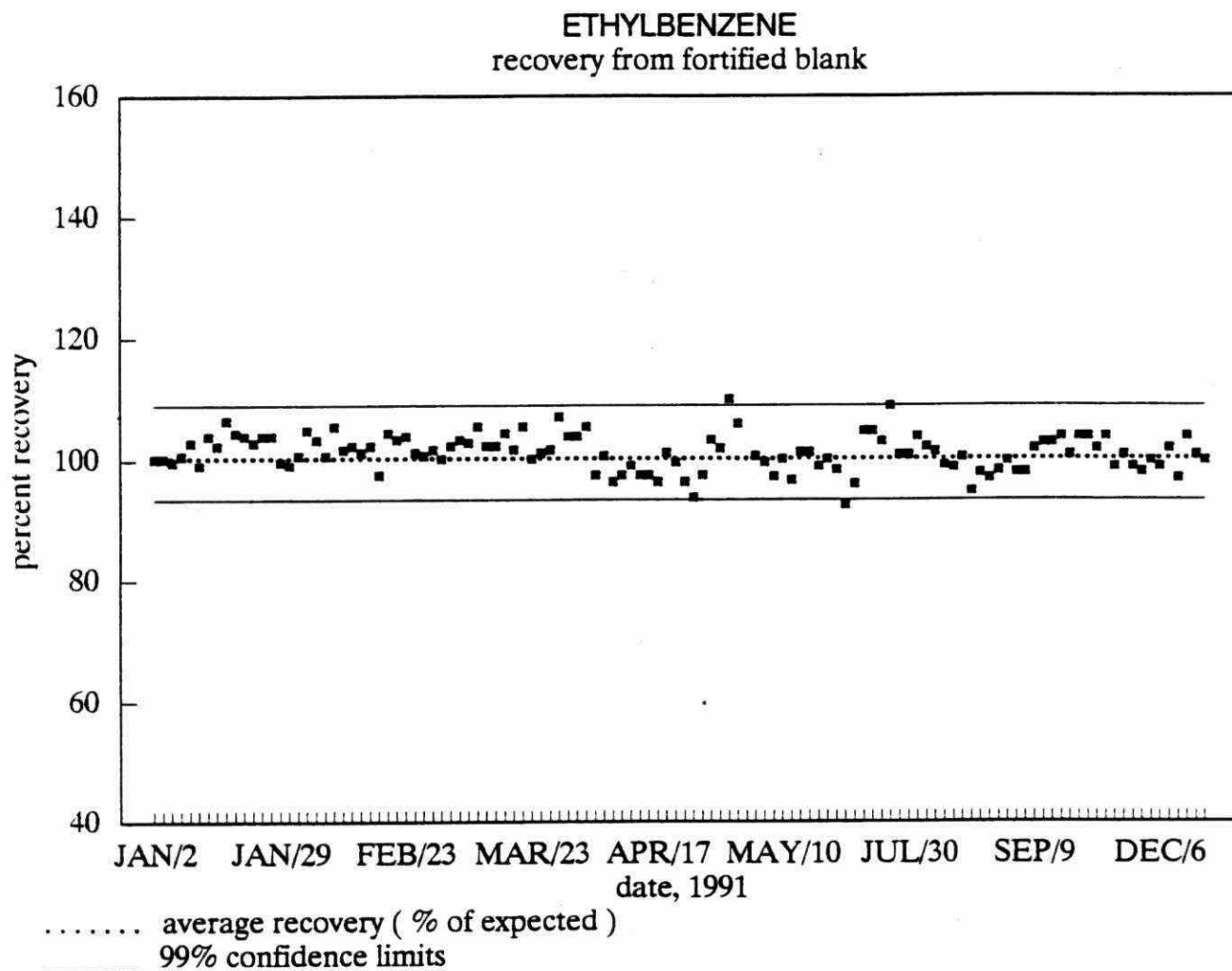
Analyte	1,2-dibromoethane
True Concentration	1.85 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.1% (n=4)
Between-run Standard Deviation	6.8%
Accuracy (% of expected)	102%



Performance Summary Table

January - December 1991

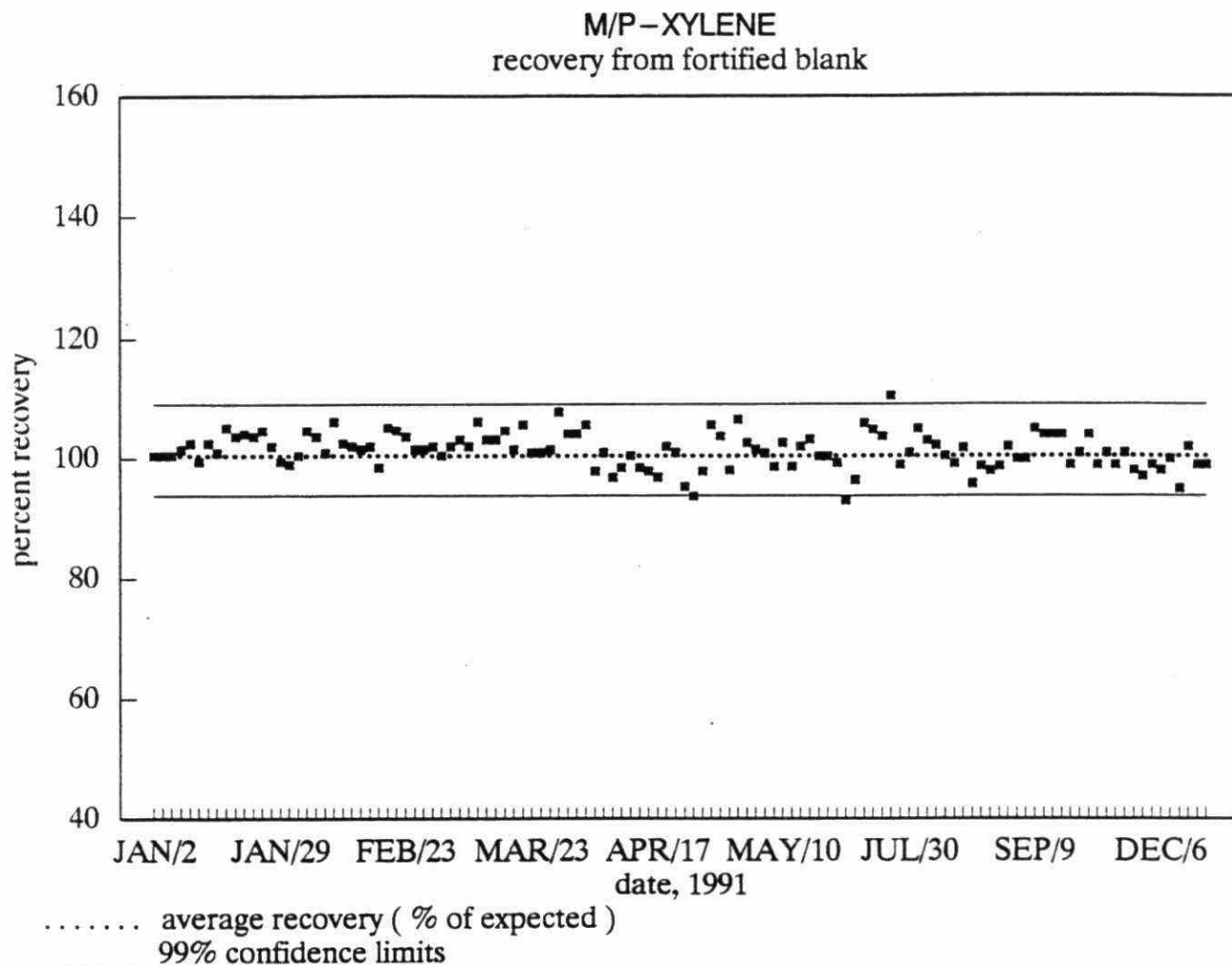
Analyte	chlorobenzene
True Concentration	1.86 µg/L, 4.6 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% (n=4)
Between-run Standard Deviation	3.3%
Accuracy (% of expected)	100.4%



Performance Summary Table

January - December 1991

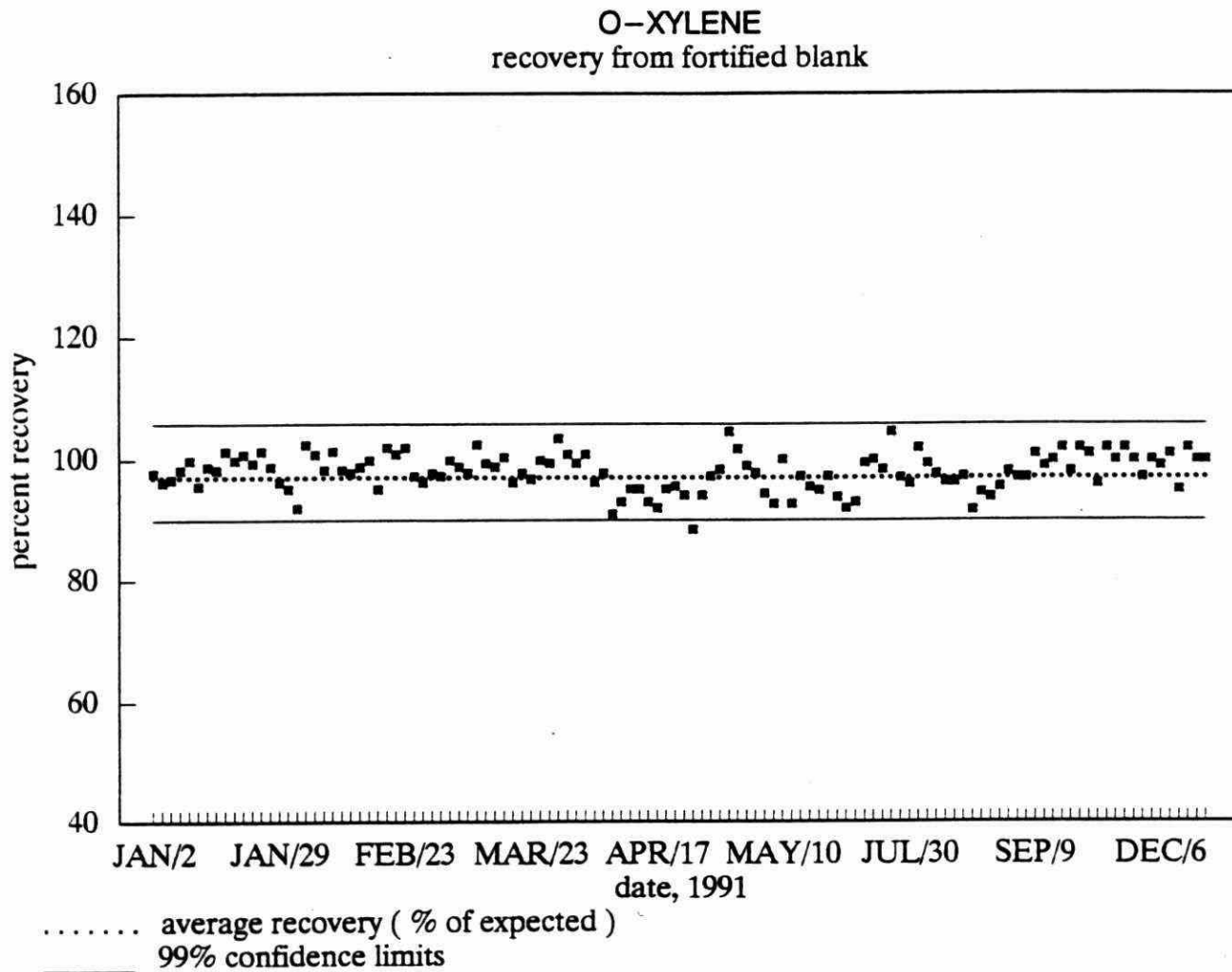
Analyte	ethylbenzene
True Concentration	1.87 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.2% (n=4)
Between-run Standard Deviation	3.0%
Accuracy (% of expected)	101%



Performance Summary Table

January - December 1991

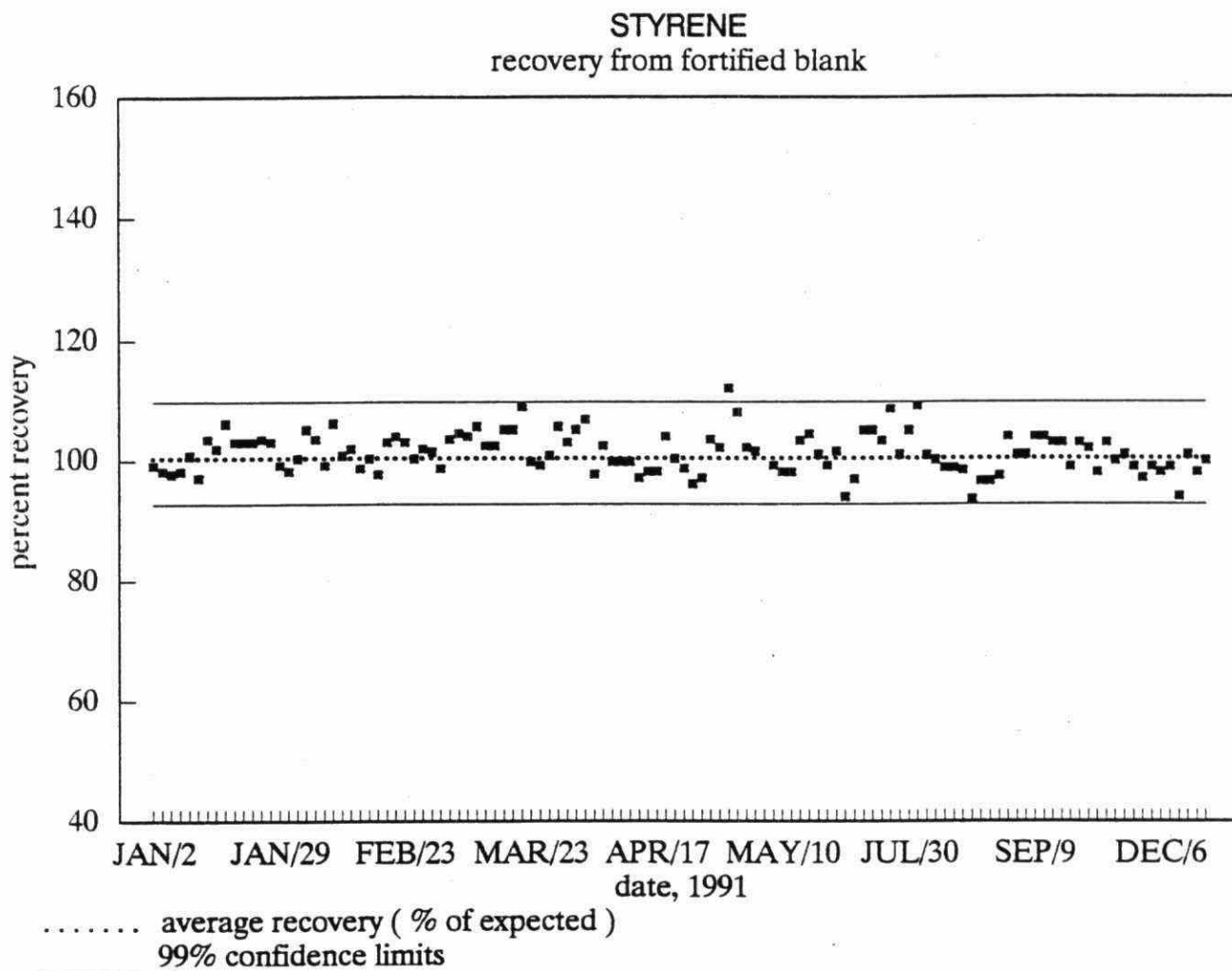
Analyte	m/p-xylene
True Concentration	1.94 µg/L, 3.68 µg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1.3% (n=4)
Between-run Standard Deviation	2.9%
Accuracy (% of expected)	101%



Performance Summary Table

January - December 1991

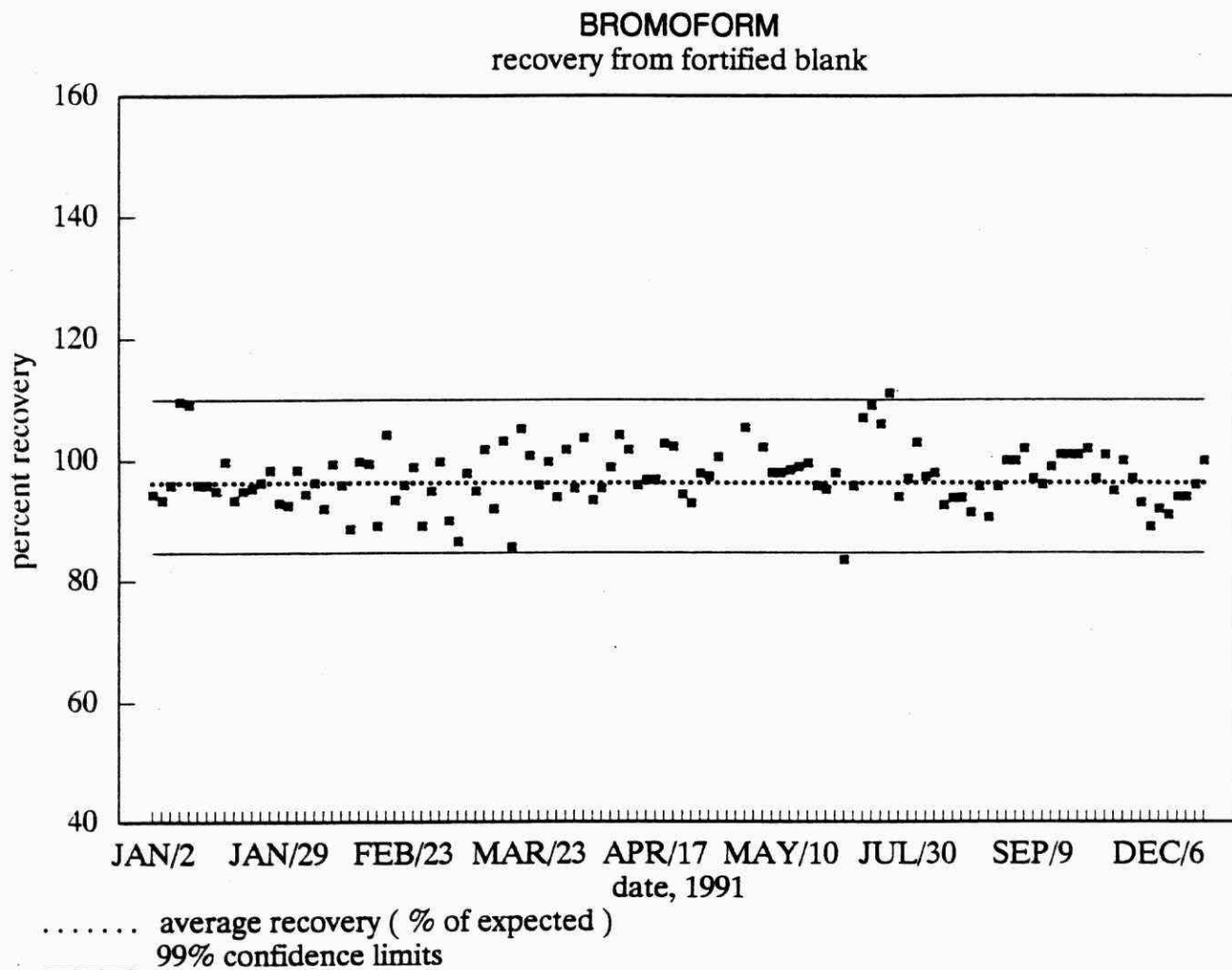
Analyte	o-xylene
True Concentration	1.91 µg/L, 3.68 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.5% (n=4)
Between-run Standard Deviation	3.1%
Accuracy (% of expected)	98%



Performance Summary Table

January - December 1991

Analyte	styrene
True Concentration	1.88 µg/L, 3.68 µg/L
Number of Observations	117
Within-run Rel. Standard Deviation	1.8% (n=4)
Between-run Standard Deviation	3.3%
Accuracy (% of expected)	101%

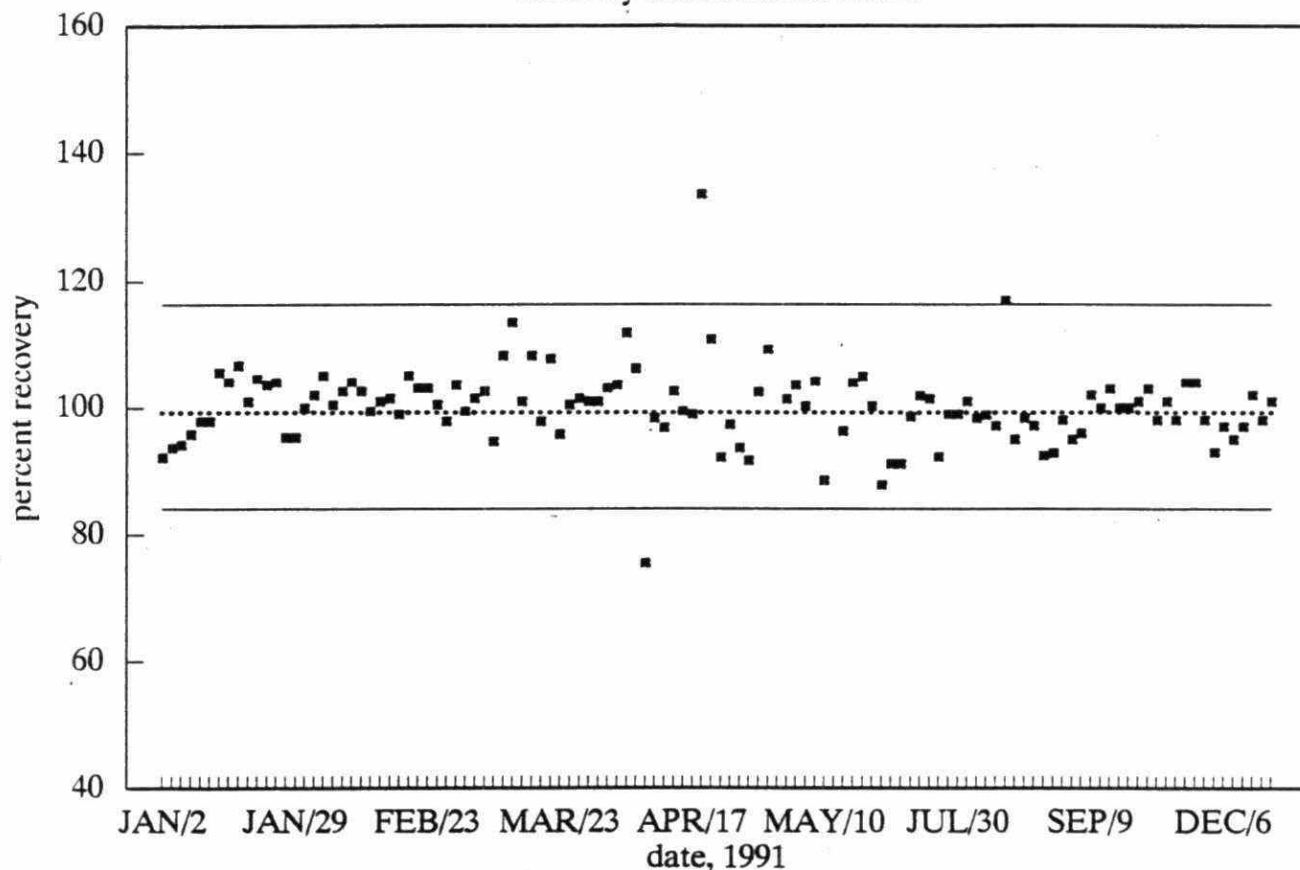


Performance Summary Table

January - December 1991

Analyte	bromoform
True Concentration	2.04 µg/L, 7.36 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	4.8% (n=4)
Between-run Standard Deviation	4.9%
Accuracy (% of expected)	97%

1,1,2,2-TETRACHLOROETHANE
recovery from fortified blank



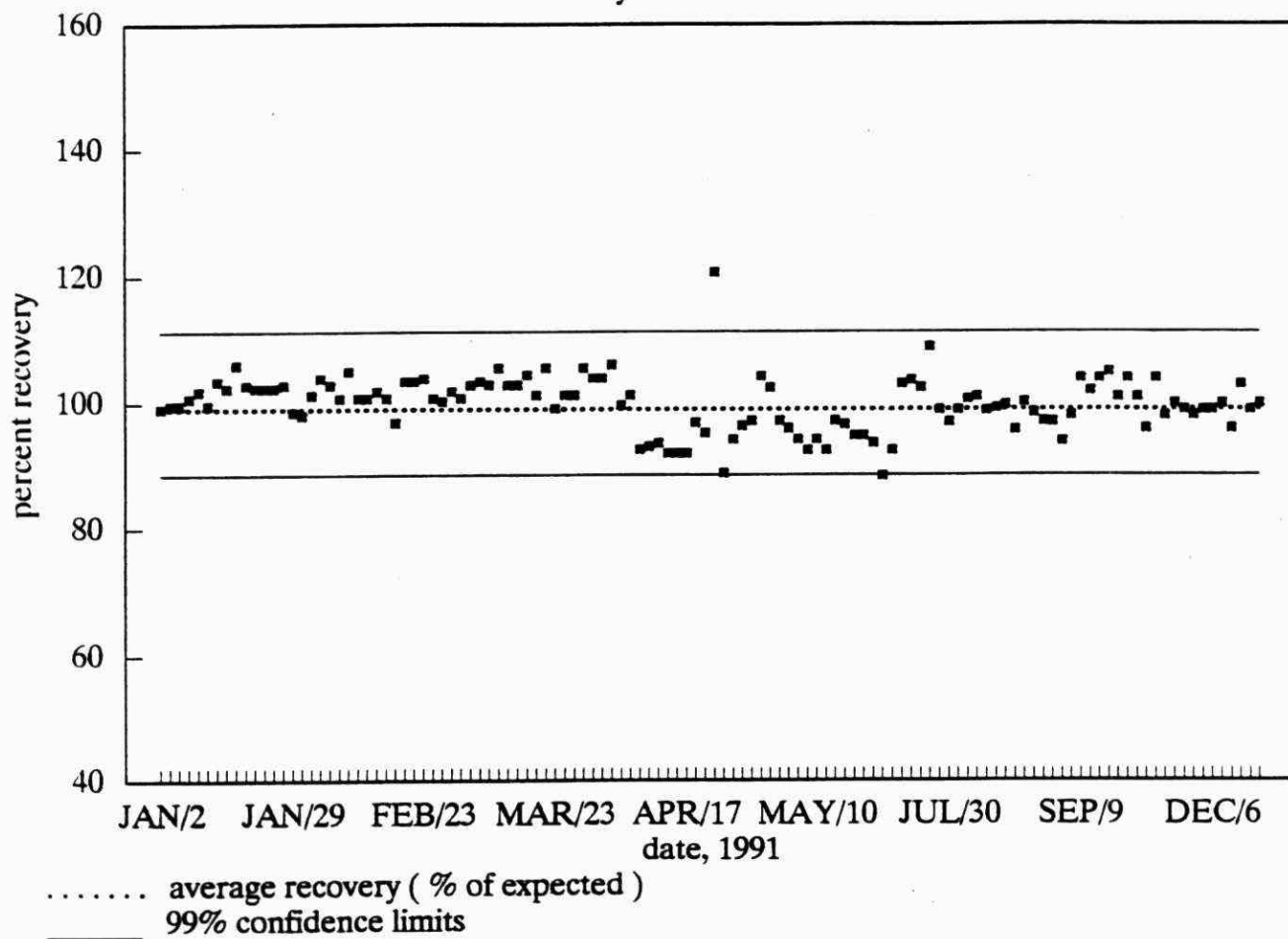
..... average recovery (% of expected)
——— 99% confidence limits

Performance Summary Table

January - December 1991

Analyte	1,1,2,2-tetrachloroethane
True Concentration	1.93 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	2.6% (n=4)
Between-run Standard Deviation	7.0%
Accuracy (% of expected)	99.5%

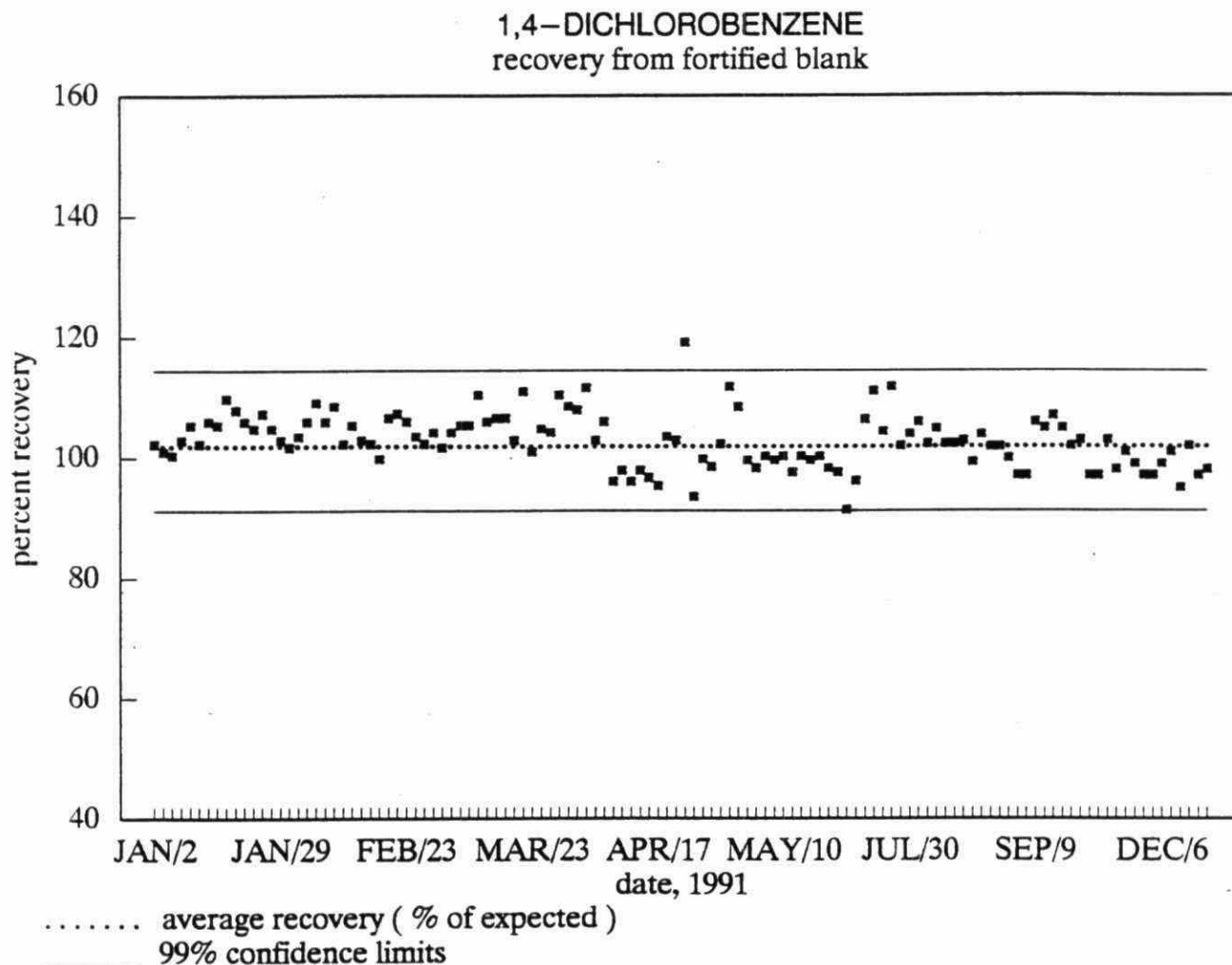
1,3-DICHLOROBENZENE
recovery from fortified blank



Performance Summary Table

January - December 1991

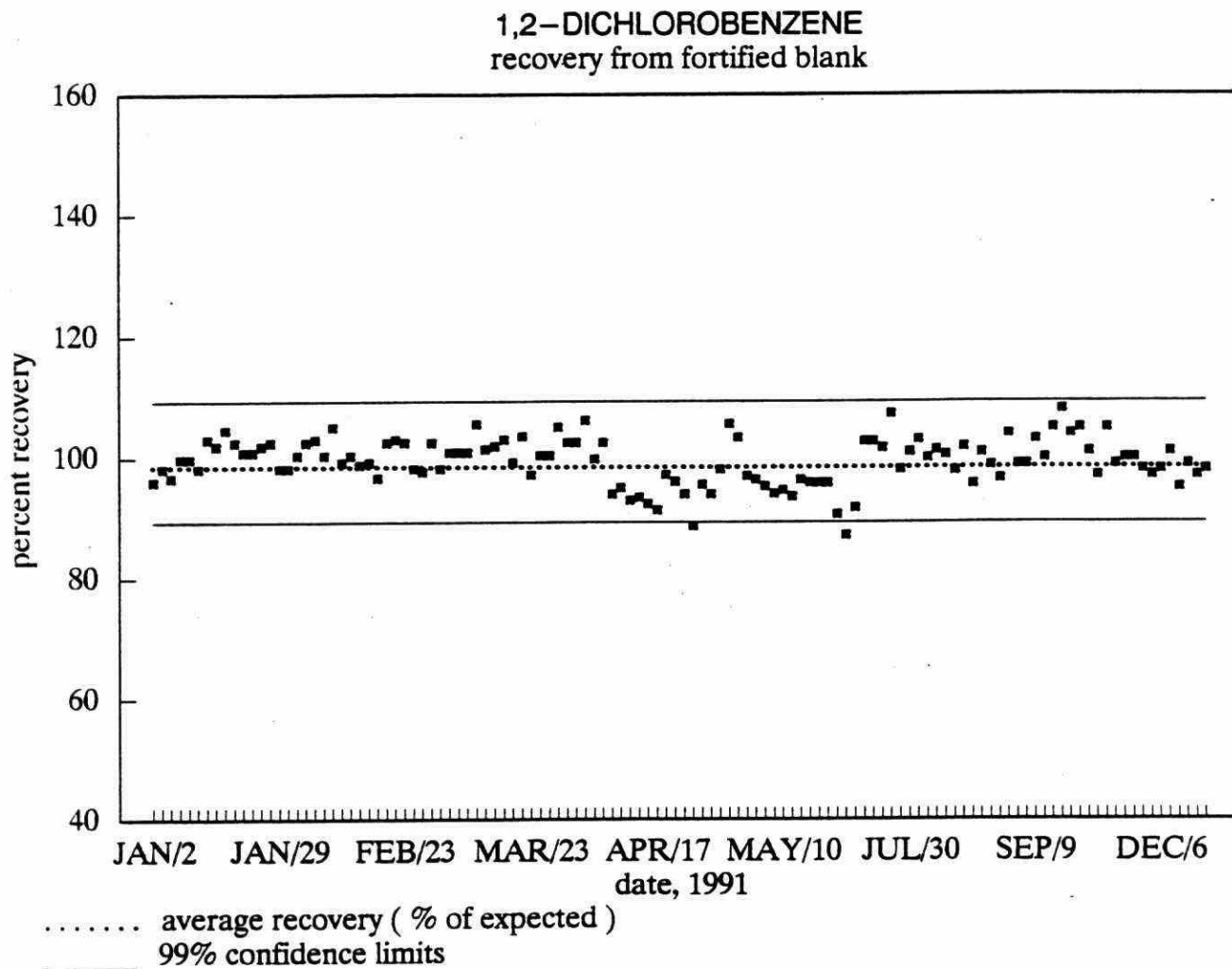
Analyte	1,3-dichlorobenzene
True Concentration	1.86 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.0% (n=4)
Between-run Standard Deviation	4.4%
Accuracy (% of expected)	100%



Performance Summary Table

January - December 1991

Analyte	1,4-dichlorobenzene
True Concentration	1.59 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	1.0% (n=4)
Between-run Standard Deviation	4.5%
Accuracy (% of expected)	103%



Performance Summary Table

January - December 1991

Analyte	1,2-dichlorobenzene
True Concentration	1.88 µg/L, 5.52 µg/L
Number of Observations	118
Within-run Rel. Standard Deviation	0.9% (n=4)
Between-run Standard Deviation	3.9%
Accuracy (% of expected)	99.3%

METHOD CODE : OWOC-E3120A.1

METHOD TITLE: The Determination of Organochlorine Pesticides, Polychlorinated Biphenyls and Other Chlorinated Organic Compounds in Water by GC-ECD

LABORATORY : Organic Water Unit

SUPERVISOR : C.D. Hall

SAMPLE TYPE : surface water, groundwater, finished drinking water

PRINCIPLE OF THE METHOD :

Samples are extracted with solvent; the extract is dried, concentrated, cleaned-up on Florisil, and reconstituted prior to analysis by dual column capillary gas chromatography with dual electron capture detection.

PARAMETERS MEASURED :	LIS TEST CODE :	W (ng/L)	T (ng/L)
hexachloroethane	X2HCE	1	10
1,3,5-trichlorobenzene	X2135	5	50
1,2,4-trichlorobenzene	X2124	5	50
1,2,3-trichlorobenzene	X2123	5	50
hexachlorobutadiene	X1HCBD	1	10
2,4,5-trichlorotoluene	X2T245	5	50
2,3,6-trichlorotoluene	X2T236	5	50
1,2,3,5-tetrachlorobenzene	X21235	1	10
1,2,4,5-tetrachlorobenzene	X21245	1	10
1,2,3,4-tetrachlorobenzene	X21234	1	10
α ,2,6-trichlorotoluene	X2T26A	5	50
pentachlorobenzene	X2PNCB	1	10
hexachlorocyclopentadiene	X1HCCP	5	50
hexachlorobenzene	X2HCB	1	10
heptachlor	P1HEPT	1	10
aldrin	P1ALDR	1	10
p,p'-DDE	P1PPDE	1	10
α -BHC	P1BHCA	1	10
β -BHC	P1BHCB	1	10
γ -BHC	P1BHCG	1	10
α -chlordane	P1CHLA	2	20
γ -chlordane	P1CHLG	2	20
oxychlordane	P1OCHL	2	20
o,p'-DDT	P1OPDT	5	50
p,p'-DDD	P1PPDD	5	50
p,p'-DDT	P1PPDT	5	50
methoxychlor	P1DMDT	5	50

(parameters measured continued)

heptachlor epoxide	P1HEPE	1	10
endosulfan I	P1END1	2	20
dieldrin	P1DIEL	2	20
endrin	P1ENDR	5	50
endosulfan II	P1END2	5	50
endosulfan cyclic sulfate	P1ENDS	5	50
mirex	P1MIRX	5	50
total PCB's	P1PCBT	20	200
octachlorostyrene	X2OCST	1	10
toxaphene	P1TOX	500	5 000

REPORTING FORMAT :

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

REMARKS : In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

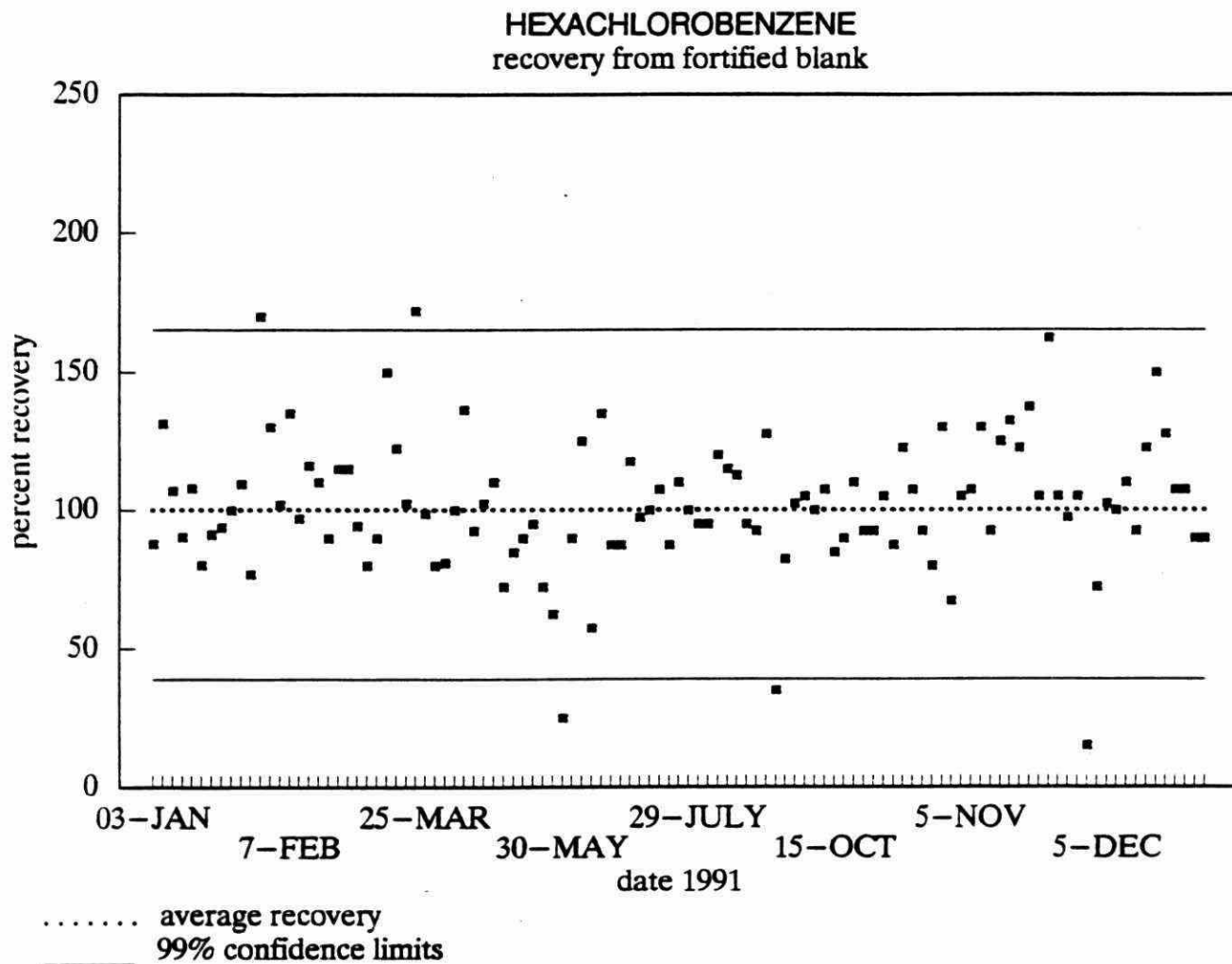
List of Performance Charts : Hexachlorobenzene (recovery from fortified blank)
1,3,5-Trichlorobenzene (recovery from fortified blank)
Hexachlorobutadiene (recovery from fortified blank)
Mirex (recovery from fortified blank)
Total PCB (recovery from fortified blank)

List of Performance Tables : Method Blanks Summary
Hexachlorobenzene
1,3,5-Trichlorobenzene
Hexachlorobutadiene
Mirex
Total PCB

Method Blanks Summary

January 1991 - December 1991

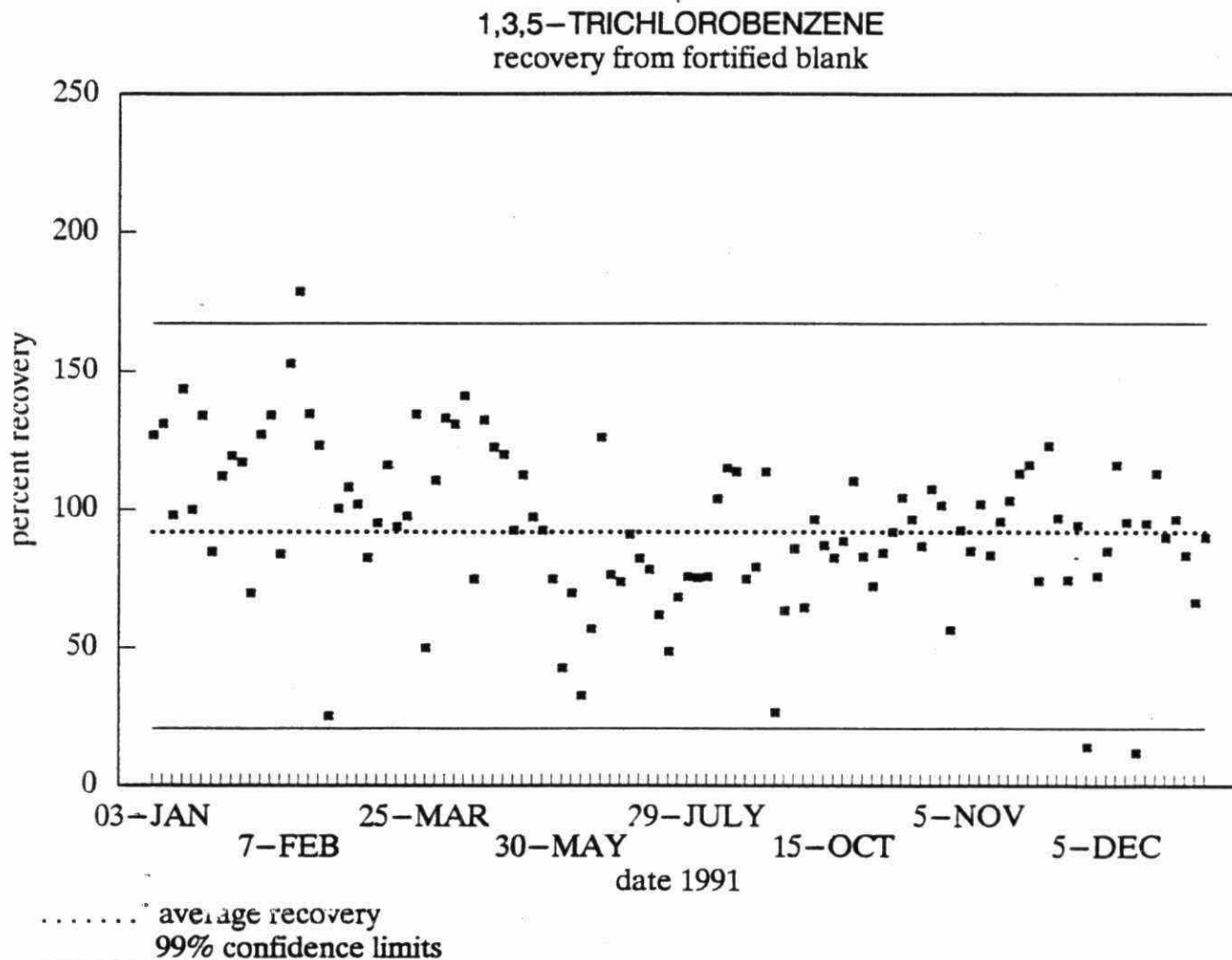
Analyte	Number of Observations	Average Concentration (ng/L)	Standard Deviation (ng/L)
hexachloroethane	136	1.5	3.6
1,3,5-trichlorobenzene	136	0	0
1,2,4-trichlorobenzene	136	0	0
1,2,3-trichlorobenzene	136	0	0
hexachlorobutadiene	136	0.04	0.25
2,4,5-trichlorotoluene	136	0.1	1.1
2,3,6-trichlorotoluene	136	0	0
1,2,3,5-tetrachlorobenzene	136	0	0
1,2,4,5-tetrachlorobenzene	136	0.04	0.051
1,2,3,4-tetrachlorobenzene	136	0.05	0.60
α ,2,6-trichlorotoluene	136	0	0
pentachlorobenzene	136	0	0
hexachlorocyclopentadiene	136	0.7	3.0
hexachlorobenzene	136	0	0
heptachlor	136	0	0
aldrin	136	0	0
p,p'-DDE	136	0.007	0.085
α -BHC	136	0.39	0.74
β -BHC	136	0.02	0.26
γ -BHC	136	0.007	0.085
α -chlordane	136	0	0
γ -chlordane	136	0	0
oxychlordane	136	0	0
o,p'-DDT	136	0	0
p,p'-DDD	136	0	0
p,p'-DDT	136	0	0
methoxychlor	136	0	0
heptachlor epoxide	136	0	0
endosulfan I	136	0	0
dieldrin	136	0	0
endosulfan II	136	0	0
endosulfan cyclic sulfate	136	0.03	0.34
mirex	136	0	0
total PCB's	136	1	13
octachlorostyrene	136	0	0
toxaphene	136	0	0



Performance Summary Table

January - December 1991

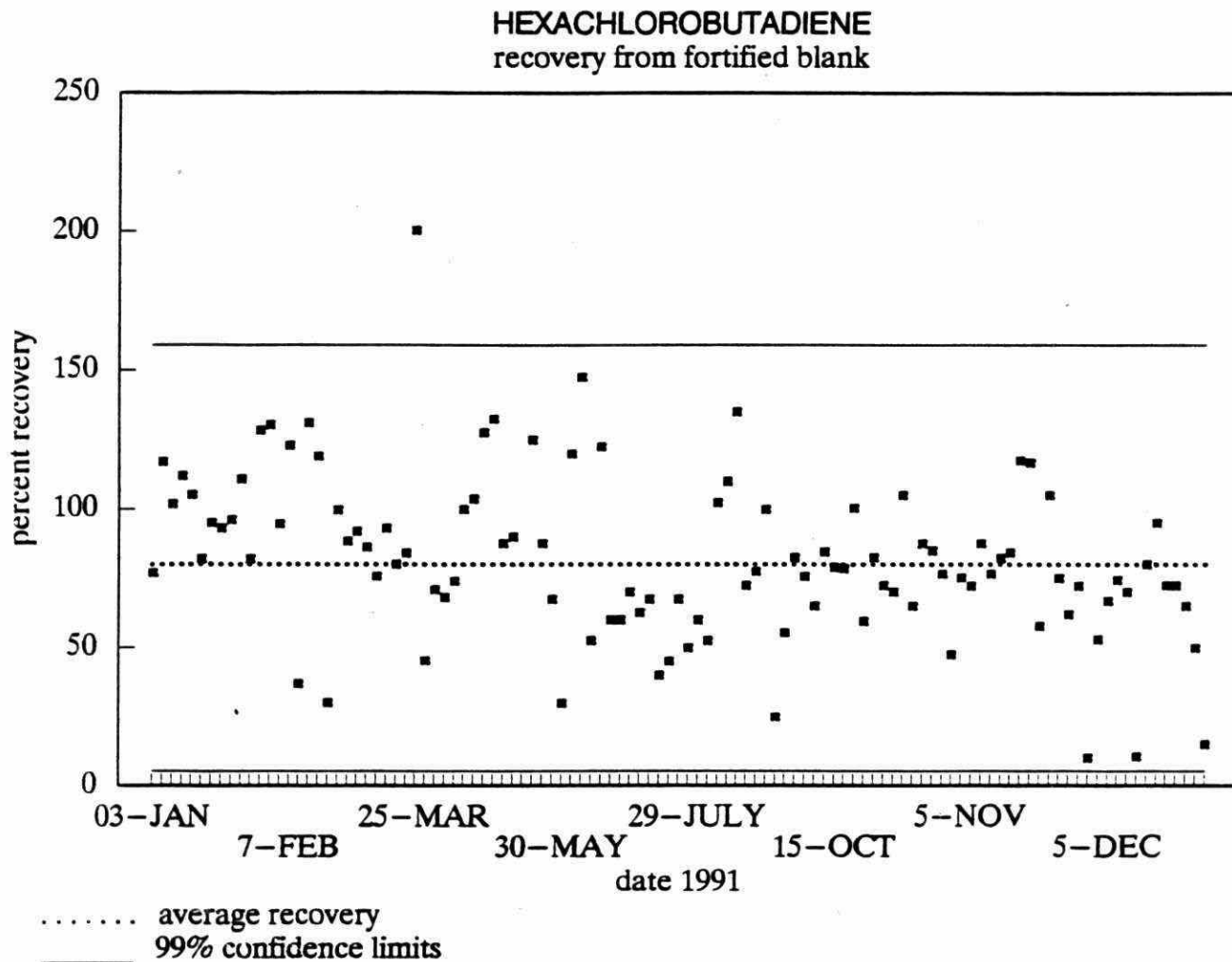
Analyte	hexachlorobenzene
True Concentration	10 ng/L; 20 ng/L
Number of Observations	109
Within-run Standard Deviation	not available
Between-run Standard Deviation	24%
Accuracy (% of expected)	102%



Performance Summary Table

January - December 1991

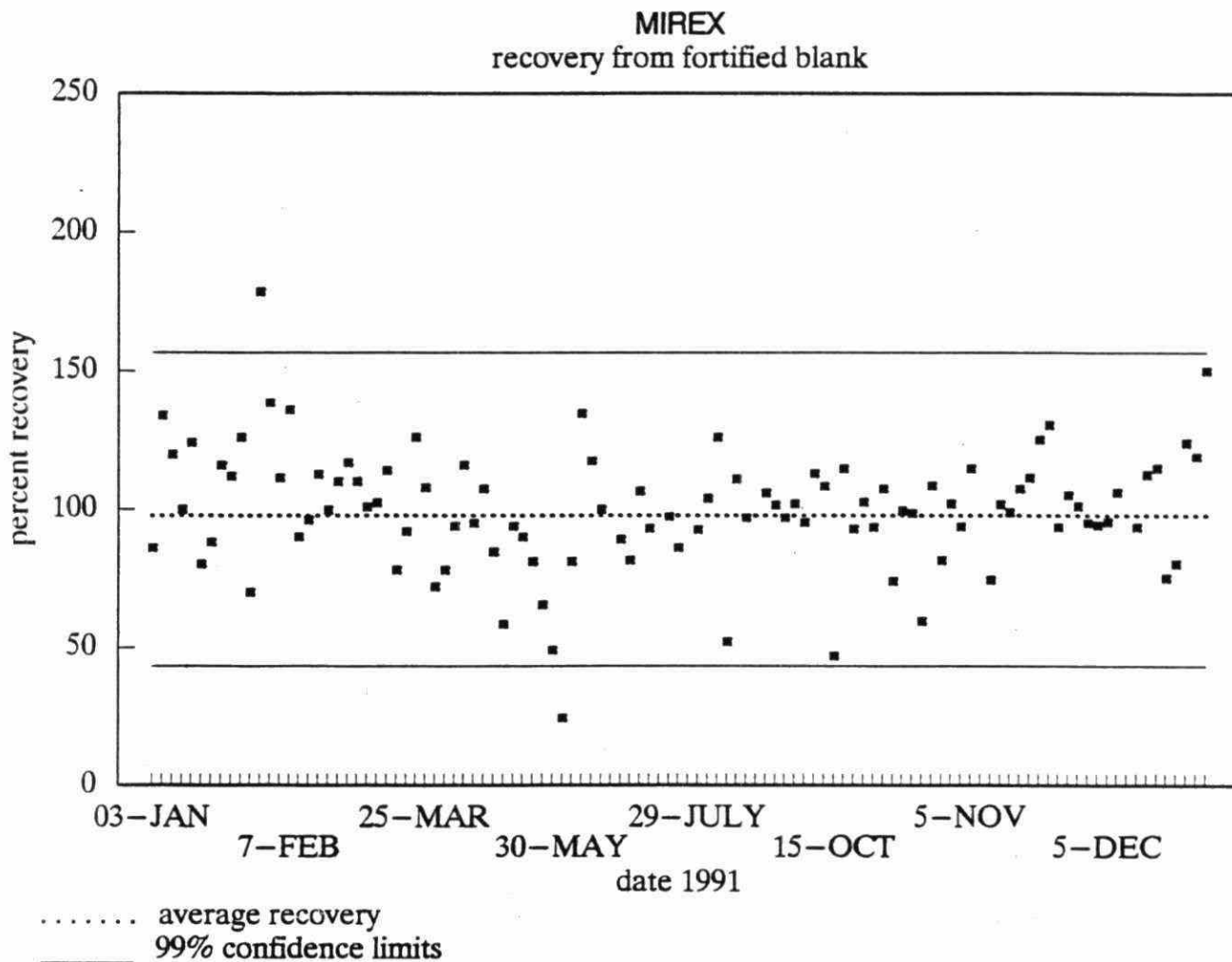
Analyte	1,3,5-trichlorobenzene
True Concentration	10 ng/L; 20 ng/L
Number of Observations	109
Within-run Standard Deviation	not available
Between-run Standard Deviation	28%
Accuracy (% of expected)	94%



Performance Summary Table

January - December 1991

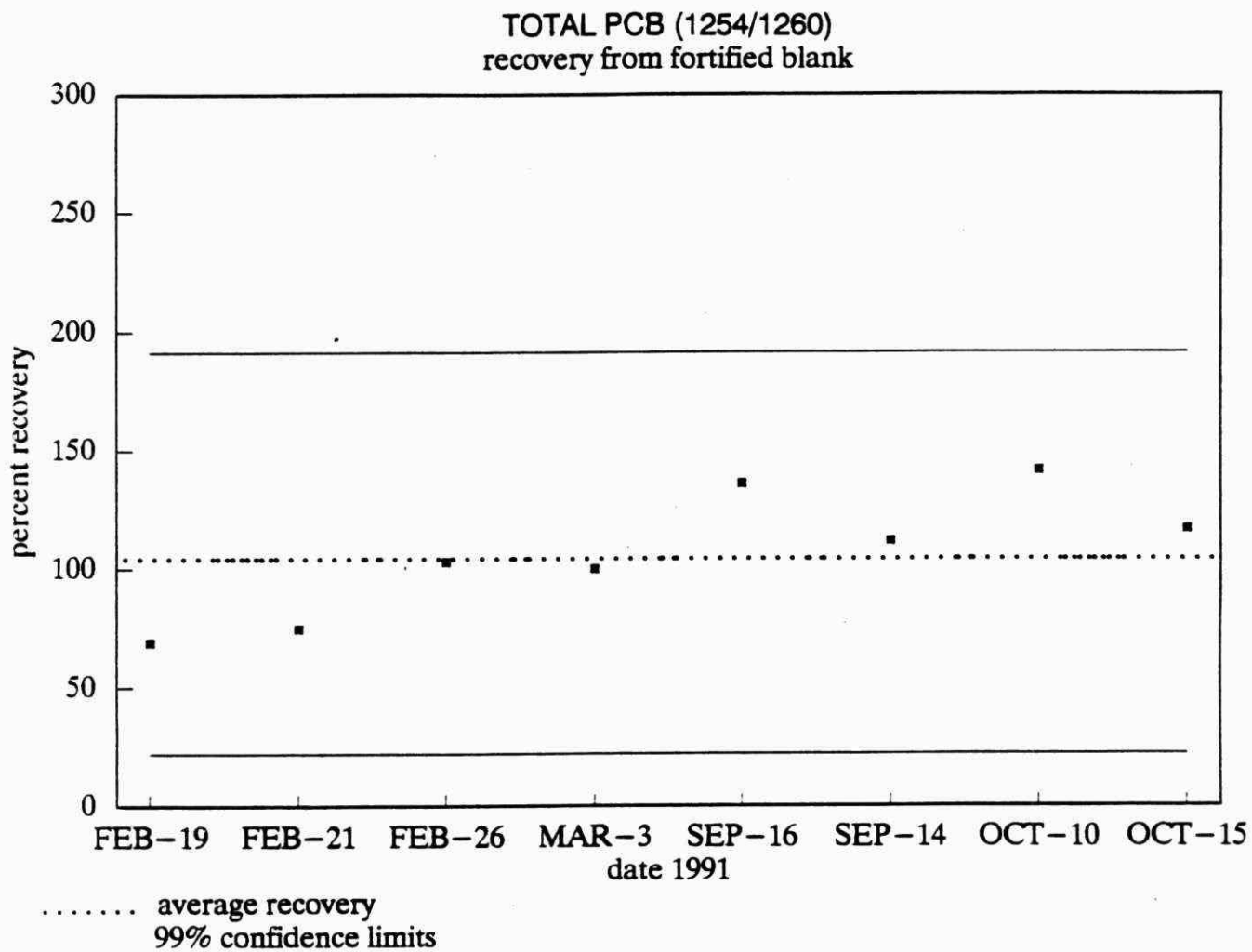
Analyte	hexachlorobutadiene
True Concentration	10 ng/L; 20 ng/L
Number of Observations	108
Within-run Standard Deviation	not available
Between-run Standard Deviation	30%
Accuracy (% of expected)	82%



Performance Summary Table

January - December 1991

Analyte	mirex
True Concentration	50 ng/L; 100 ng/L
Number of Observations	105
Within-run Standard Deviation	not available
Between-run Standard Deviation	22%
Accuracy (% of expected)	98%



Performance Summary Table

January - December 1991

Analyte	total PCB (1254/1260)
True Concentration	200 ng/L
Number of Observations	8
Within-run Standard Deviation	not available
Between-run Standard Deviation	24%
Accuracy (% of expected)	107%

METHOD CODE : OWCP-B-E3119A.1

METHOD TITLE: The Determination of Chlorophenols and Phenoxyacid Herbicides in Water by Using Solid Phase Extraction and GC-ECD

LABORATORY : Organic Water Unit

SUPERVISOR : C.D. Hall

SAMPLE TYPE : surface water, groundwater, finished drinking water

PRINCIPLE OF THE METHOD :

The aqueous sample is aspirated through C-18 bonded porous silica cartridge. The cartridge is eluted with a small volume of solvent. The eluate is methylated and the 2,4-D type herbicides and chlorophenols are determined as the corresponding methyl esters and ethers by dual capillary gas chromatography with electron capture detection.

PARAMETERS MEASURED :	LIS TEST CODE :	W (ng/L)	T (ng/L)
2,4,6-trichlorophenol	X3246	20	200
2,4,5-trichlorophenol	X3245	100	1 000
2,3,4-trichlorophenol	X3234	100	1 000
2,3,5,6-tetrachlorophenol	X32356	10	100
2,3,4,5-tetrachlorophenol	X32345	20	200
pentachlorophenol	X3PCPH	10	100
Dicamba	P3DICA	50	500
2,4-dichlorophenoxypropanoic acid	P324DP	100	1 000
2,4-dichlorophenoxyacetic acid	P324D	100	1 000
Silvex	P3SILV	20	200
2,4,5-trichlorophenoxyacetic acid	P3245T	50	500
2,4-dichlorophenoxybutyric acid	P324DB	200	2 000
Picloram	P3PICL	100	1 000

REPORTING FORMAT :

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

QUALITY CONTROL :

Quality control samples included in the run format are method blanks, fortified method blanks and calibration check solution.

For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

REMARKS : In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

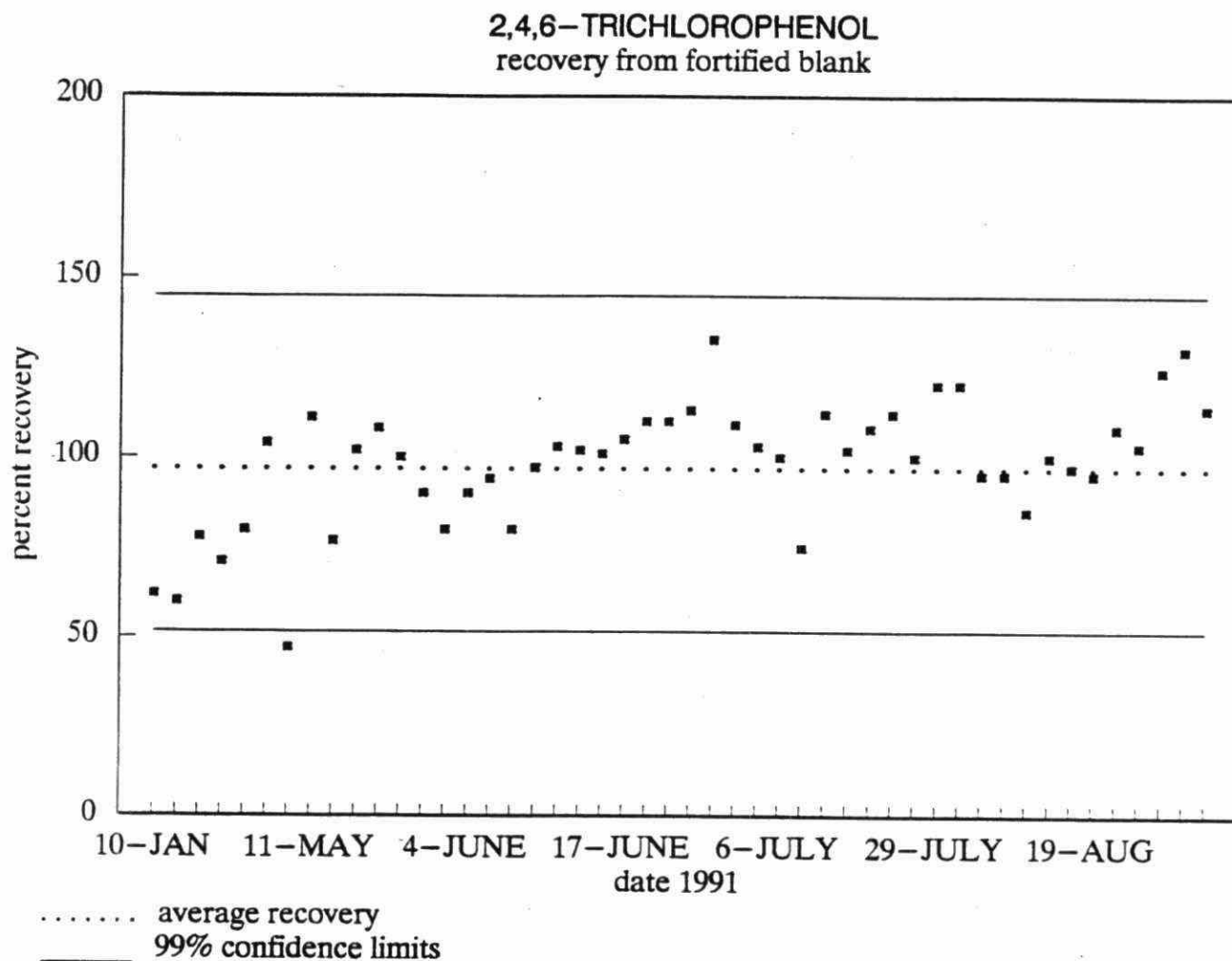
List of Performance Charts : 2,4,6-Trichlorophenol (recovery from fortified blank)
2,4-Dichlorophenoxyacetic Acid (recovery from fortified blank)
Silvex (recovery from fortified blank)

List of Performance Tables : Method Blanks Summary
2,4,6-Trichlorophenol
2,4-Dichlorophenoxyacetic Acid
Silvex

Method Blanks Summary

January 1991 - December 1991

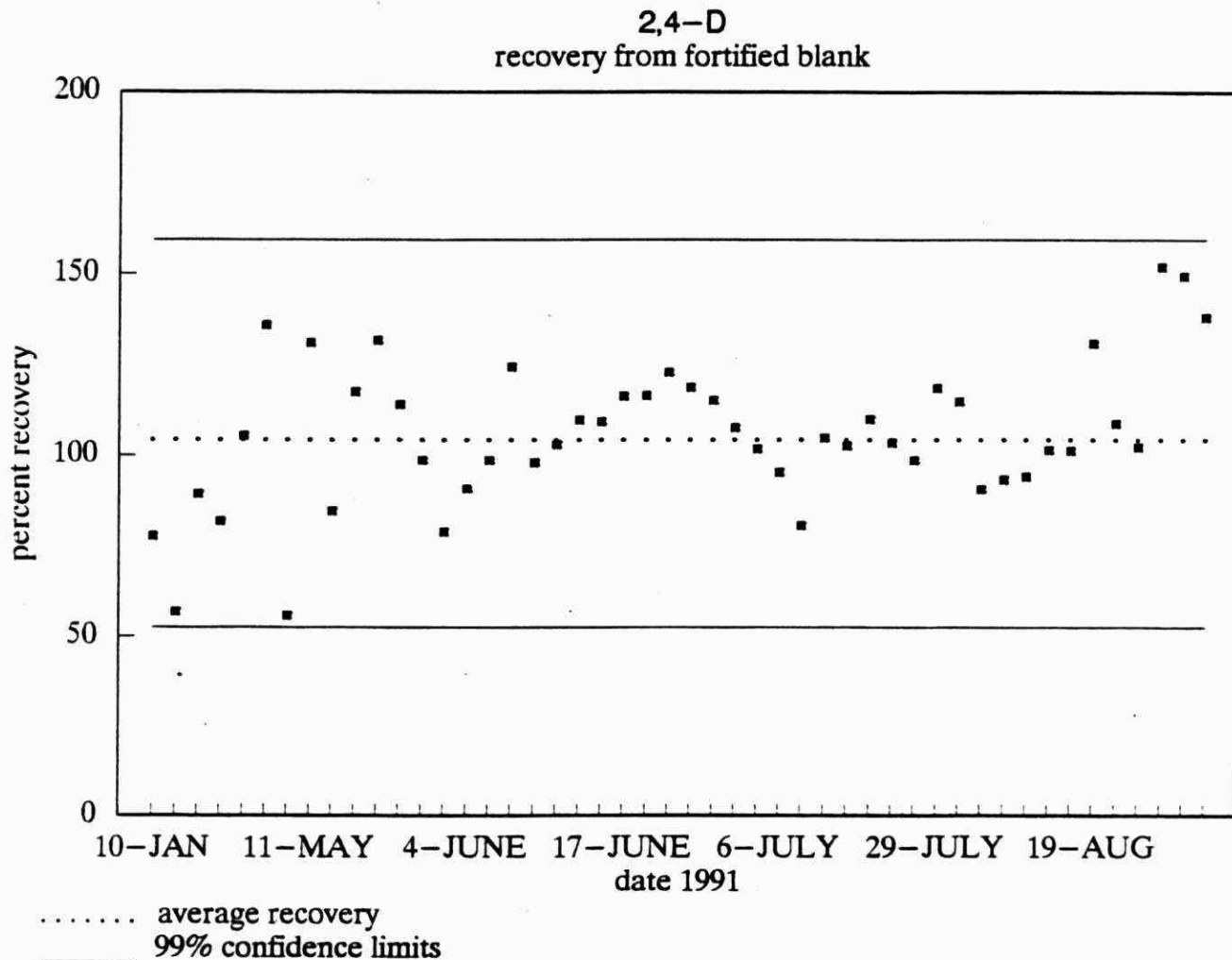
Analyte	Number of Observations	Average Concentration (ng/L)	Standard Deviation (ng/L)
2,4,6-trichlorophenol	24	0	0
2,4,5-trichlorophenol	24	0	0
2,3,4-trichlorophenol	24	0	0
2,3,5,6-tetrachlorophenol	24	0	0
2,3,4,5-tetrachlorophenol	24	0	0
pentachlorophenol	24	6	42
Dicamba	24	0	0
2,4-dichlorophenoxypropanoic acid	24	0	0
2,4-dichlorophenoxyacetic acid	24	0	0
Silvex	24	0	0
2,4,5-trichlorophenoxyacetic acid	24	0	0
2,4-dichlorophenoxybutyric acid	24	0	0
Picloram	24	0	0



Performance Summary Table

January - December 1991

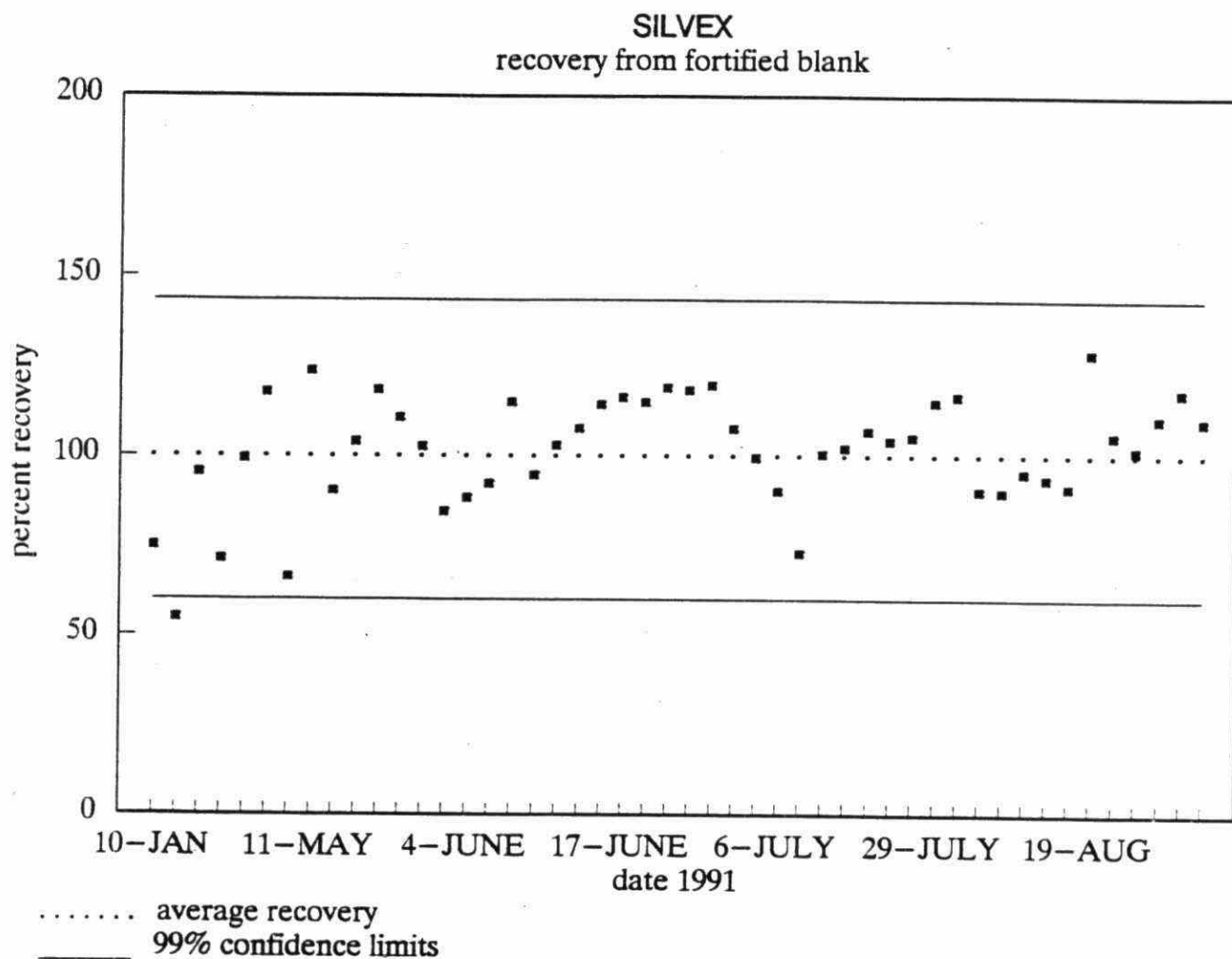
Analyte	2,4,6-trichlorophenol
True Concentration	100 ng/L
Number of Observations	48
Within-run Standard Deviation	not available
Between-run Standard Deviation	17%
Accuracy (% of expected)	98%



Performance Summary Table

January - December 1991

Analyte	2,4-D (2,4-dichlorophenoxyacetic acid)
True Concentration	750 ng/L
Number of Observations	40
Within-run Standard Deviation	not available
Between-run Standard Deviation	20%
Accuracy (% of expected)	105%



Performance Summary Table

January - December 1991

Analyte	Silvex
True Concentration	150 ng/L
Number of Observations	48
Within-run Standard Deviation	not available
Between-run Standard Deviation	16%
Accuracy (% of expected)	102%

METHOD CODE : PWAOP-E32224A.1
METHOD TITLE: The Determination of Organophosphorous Pesticides in Drinking Water by GC-TSD

LABORATORY : Organic Water Unit
SUPERVISOR : C.D. Hall

SAMPLE TYPE : surface water, groundwater, finished drinking water

PRINCIPLE OF THE METHOD :

Samples are solvent-extracted; water is removed from extract and extract is evaporated to dryness. The reconstituted extract is examined by dual capillary gas chromatography with a thermionic specific detector.

PARAMETERS MEASURED :	LIS TEST CODE :	W (ng/L)	T (ng/L)
methyl trithion	P4MTRI	200	2 000
dichlorvos	P4DICH	20	200
mevinphos	P4MEVI	20	200
phorate (thimet)	P4PHOR	20	200
diazinon	P4DIAZ	20	200
ronnel	P4RONN	20	200
chlorpyrifos (dursban)	P4DURS	20	200
reldan	P4RELD	20	200
malathion	P4MALA	20	200
parathion	P4PARA	20	200
methyl parathion	P4MPAR	20	200
ethion	P4ETHI	20	200

REPORTING FORMAT :

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

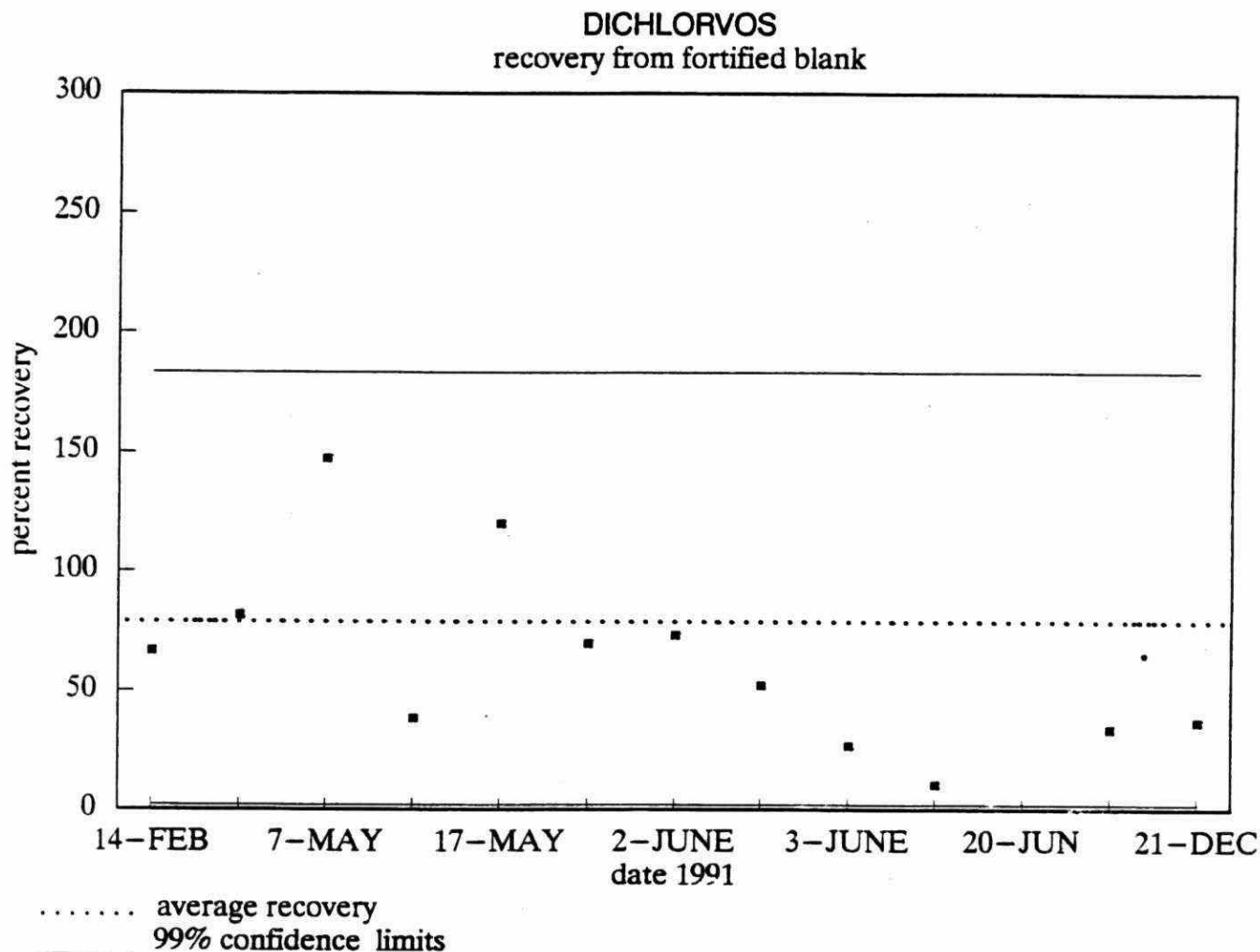
For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

REMARKS : During the period starting January 1991 and ending December 1991, a total of 24 method blanks was prepared and tested by the method. For these 24 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts : Dichlorvos (recovery from fortified blank)
 Diazinon (recovery from fortified blank)
 Ethion (recovery from fortified blank)

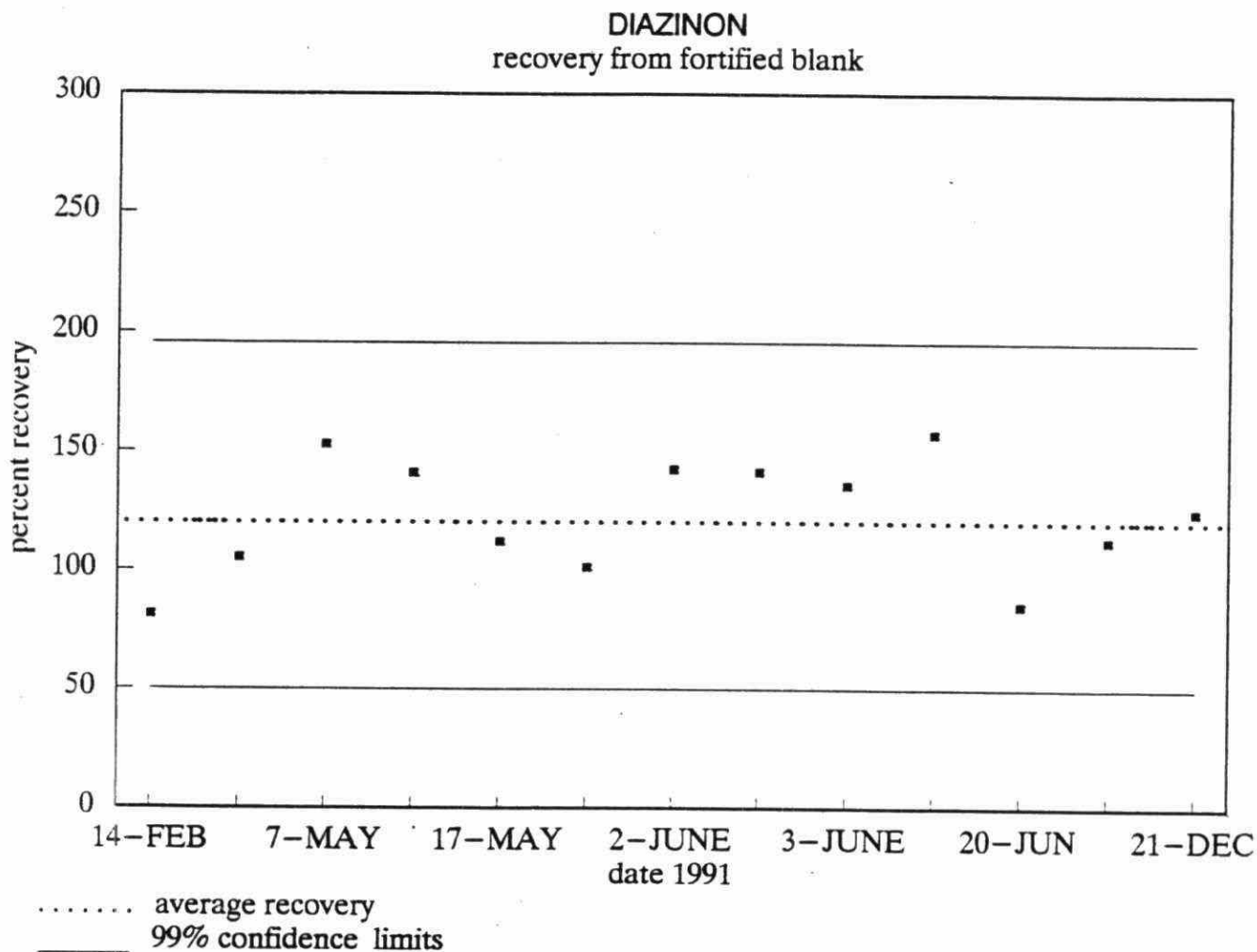
List of Performance Tables : Dichlorvos
 Diazinon
 Ethion



Performance Summary Table

January - December 1991

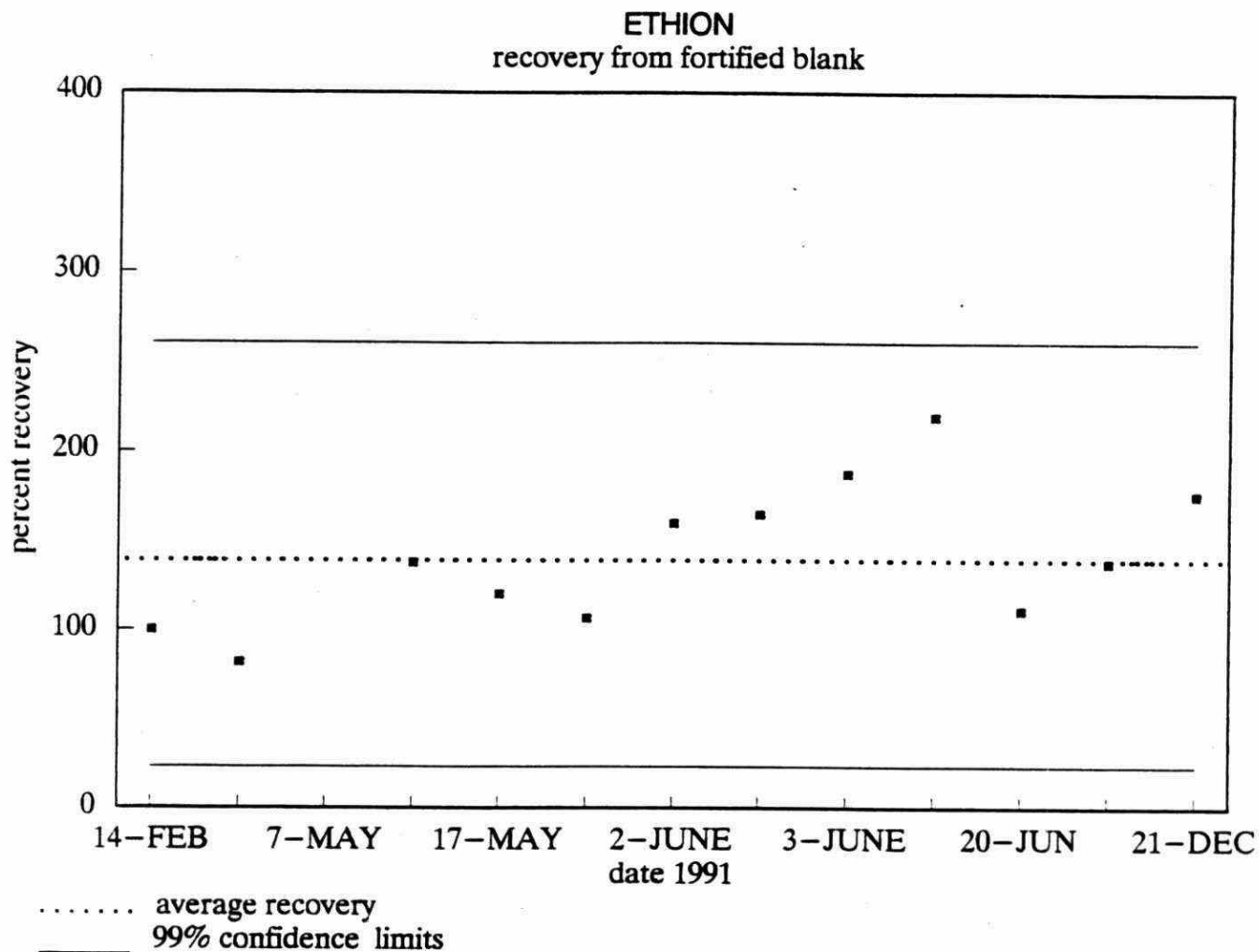
Analyte	Dichlorvos
True Concentration	100 ng/L
Number of Observations	12
Within-run Standard Deviation	not available
Between-run Standard Deviation	33%
Accuracy (% of expected)	81%



Performance Summary Table

January - December 1991

Analyte	Diazinon
True Concentration	100 ng/L
Number of Observations	13
Within-run Standard Deviation	not available
Between-run Standard Deviation	23%
Accuracy (% of expected)	122%



Performance Summary Table

January - December 1991

Analyte	Ethion
True Concentration	200 ng/L
Number of Observations	13
Within-run Standard Deviation	not available
Between-run Standard Deviation	39%
Accuracy (% of expected)	141%

METHOD CODE : HPLC/L-E3086A.1
METHOD TITLE: The Determination of Polynuclear Aromatic Hydrocarbons in Surface Water, Drinking Water and Groundwater by HPLC

LABORATORY : Organic Water Unit
SUPERVISOR : C.D. Hall

SAMPLE TYPE : surface water, groundwater, drinking water

PRINCIPLE OF THE METHOD :

Sample is solvent-extracted; the extract is dried and evaporated to dryness. The reconstituted extract is examined by high performance liquid chromatography equipped with fluorescence detector.

PARAMETERS MEASURED :	LIS TEST CODE :	W (ng/L)	T (ng/L)
phenanthrene	B3001X	10	100
anthracene	B3002X	1	10
fluoranthene	B3003X	20	200
pyrene	B3004X	20	200
benzo(a)anthracene	B3005X	20	200
chrysene	B3006X	50	500
dimethylbenz(a)anthracene	B3007X	5	50
benzo(e)pyrene	B3008X	50	500
benzo(b)fluoranthene	B3010X	10	100
perylene	B3011X	10	100
benzo(k)fluoranthene	B3012X	1	10
benzo(a)pyrene	B3013X	5	50
benzo(g,h,i)perylene	B3014X	20	200
dibenzo(a,h)anthracene	B3015X	10	100
indeno(1,2,3-c,d)pyrene	B3016X	20	200
benzo(b)chrysene	B3017X	2	20
coronene	B3019X	10	100

REPORTING FORMAT :

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W and up to maximum of two significant figures.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

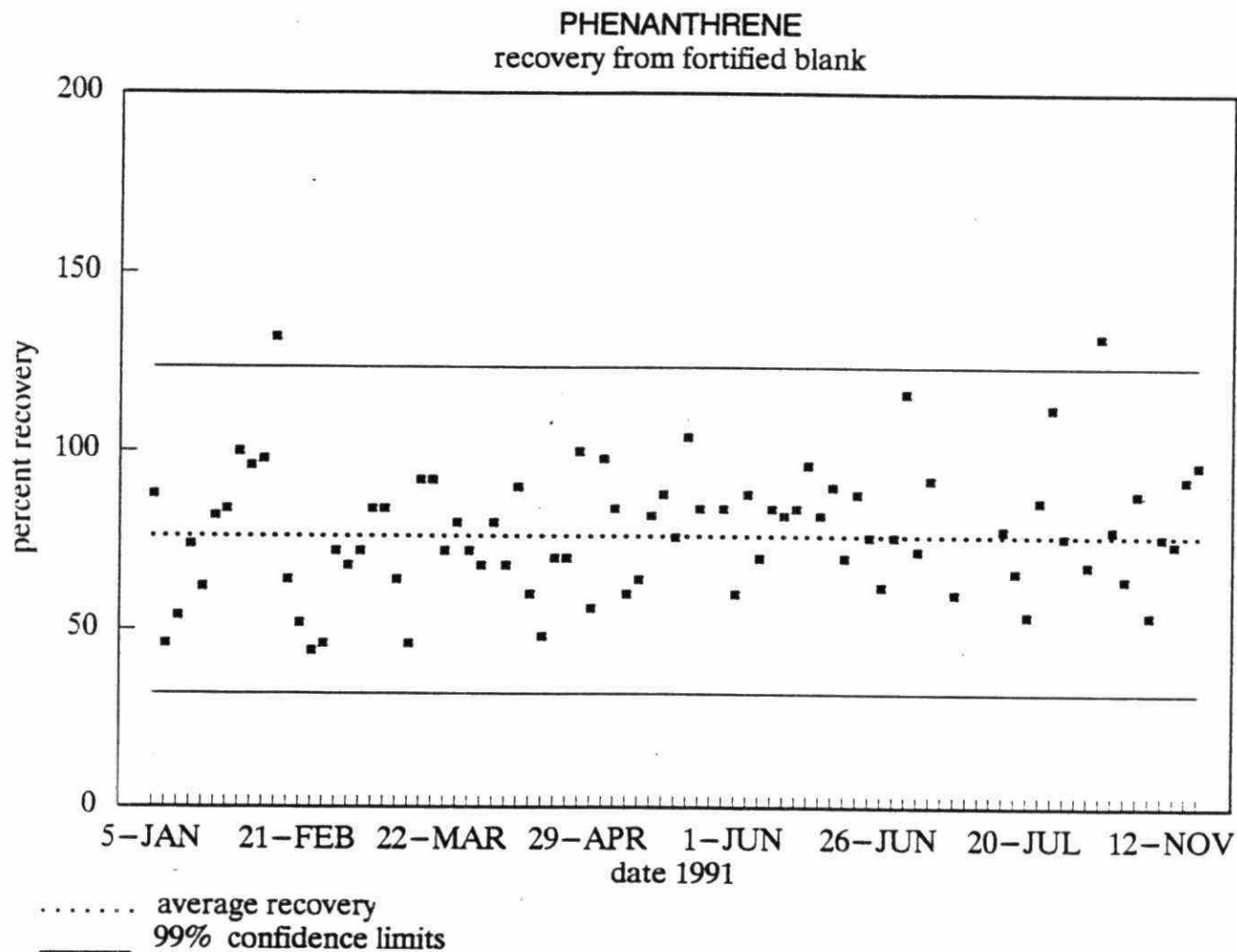
For selected target compounds, control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained.

REMARKS : During the period starting January 1991 and ending December 1991, a total of 91 method blanks was prepared and tested by the method. For these 91 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts : Phenanthrene (recovery from fortified blank)
Benzo(b)fluoranthene / Perylene (recovery from fortified blank)
Benzo(a)pyrene (recovery from fortified blank)

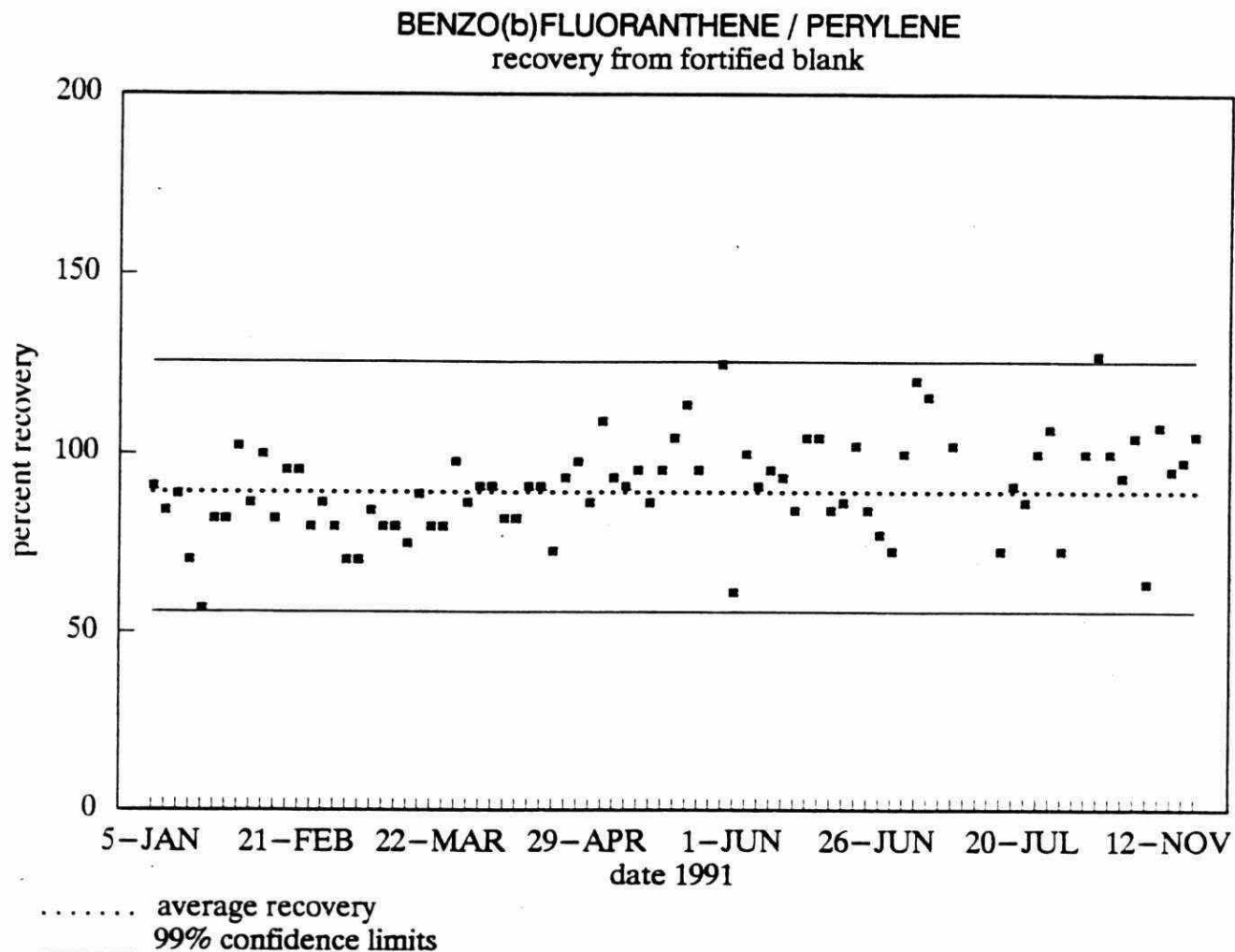
List of Performance Tables : Phenanthrene
Benzo(b)fluoranthene / Perylene
Benzo(a)pyrene



Performance Summary Table

January - December 1991

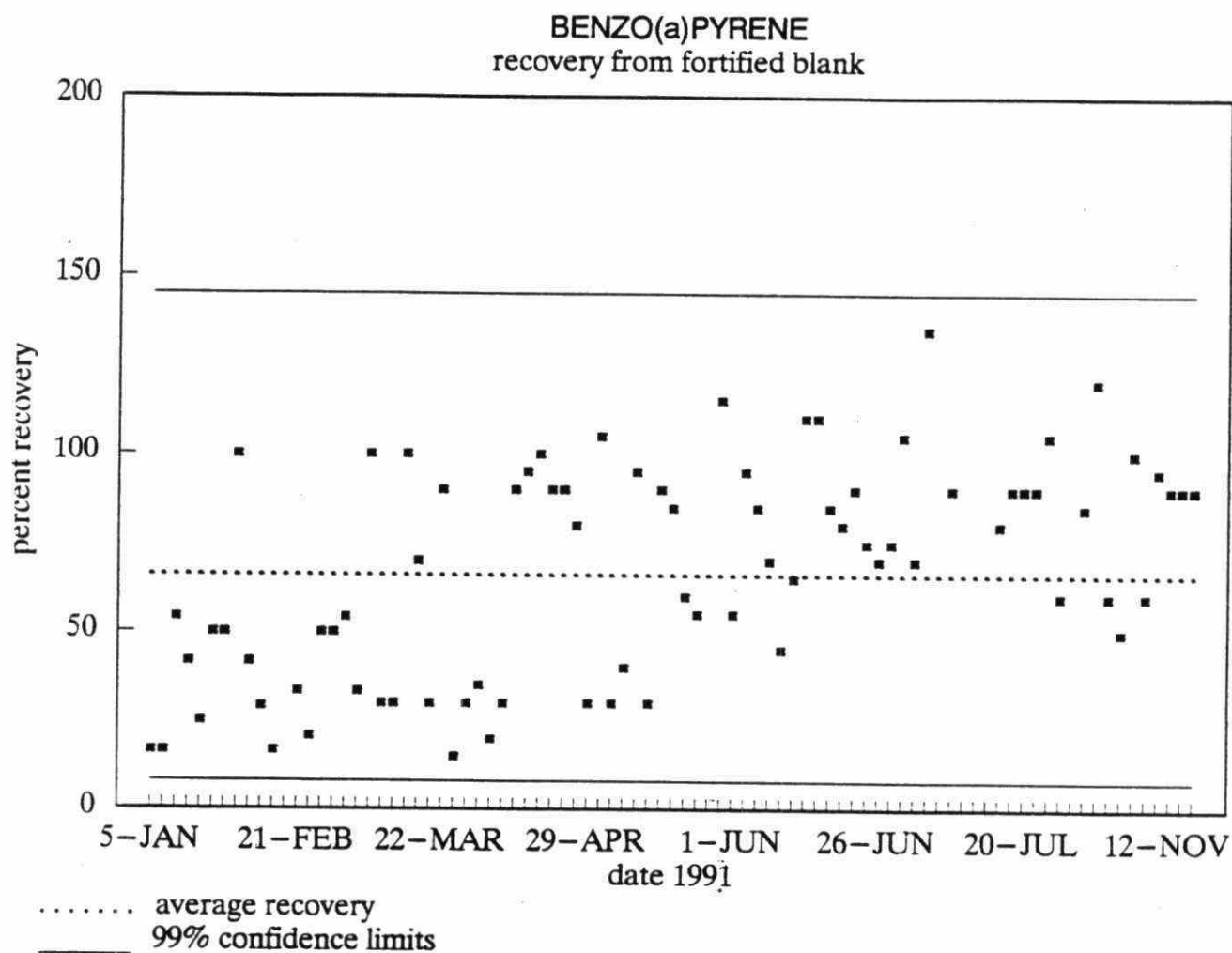
Analyte	phenanthrene
True Concentration	50 ng/L
Number of Observations	81
Within-run Standard Deviation	not available
Between-run Standard Deviation	18%
Accuracy (% of expected)	78%



Performance Summary Table

January - December 1991

Analyte	benzo(b)fluoranthene/peryene
True Concentration	44 ng/L; 40 ng/L
Number of Observations	81
Within-run Standard Deviation	not available
Between-run Standard Deviation	13%
Accuracy (% of expected)	90%



Performance Summary Table

January - December 1991

Analyte	benzo(a)pyrene
True Concentration	24 ng/L; 20ng/L
Number of Observations	80
Within-run Standard Deviation	not available
Between-run Standard Deviation	30%
Accuracy (% of expected)	68%

METHOD CODE : PWACAR-E3158A.1
METHOD TITLE: The Determination of Carbamates in Water by HPLC
LABORATORY : Organic Water Unit
SUPERVISOR : C.D. Hall
SAMPLE TYPE : surface water, groundwater, drinking water

PRINCIPLE OF THE METHOD :

Sample is solvent-extracted; the extract is dried and evaporated to dryness. The reconstituted extract is examined by high performance liquid chromatography, using a variable wavelength ultraviolet detector.

PARAMETERS MEASURED :	LIS TEST CODE :	W (ng/L)	T (ng/L)
carbofuran	P6CARB	2 000	20 000
carbaryl	P6SEVN	200	2 000
butylate	P6SUTN	2 000	20 000
propoxur	P6PROP	2 000	20 000
isopropyl-3-chlorophenyl carbamate	P6CIPC	2 000	20 000
isopropyl phenyl carbamate	P6IPC	2 000	20 000
bux	P6BUX	200	2 000
diallate	P6DIAL	2 000	20 000
eptam	P6EPTM	2 000	20 000

REPORTING FORMAT :

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W up to maximum of two significant figures.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

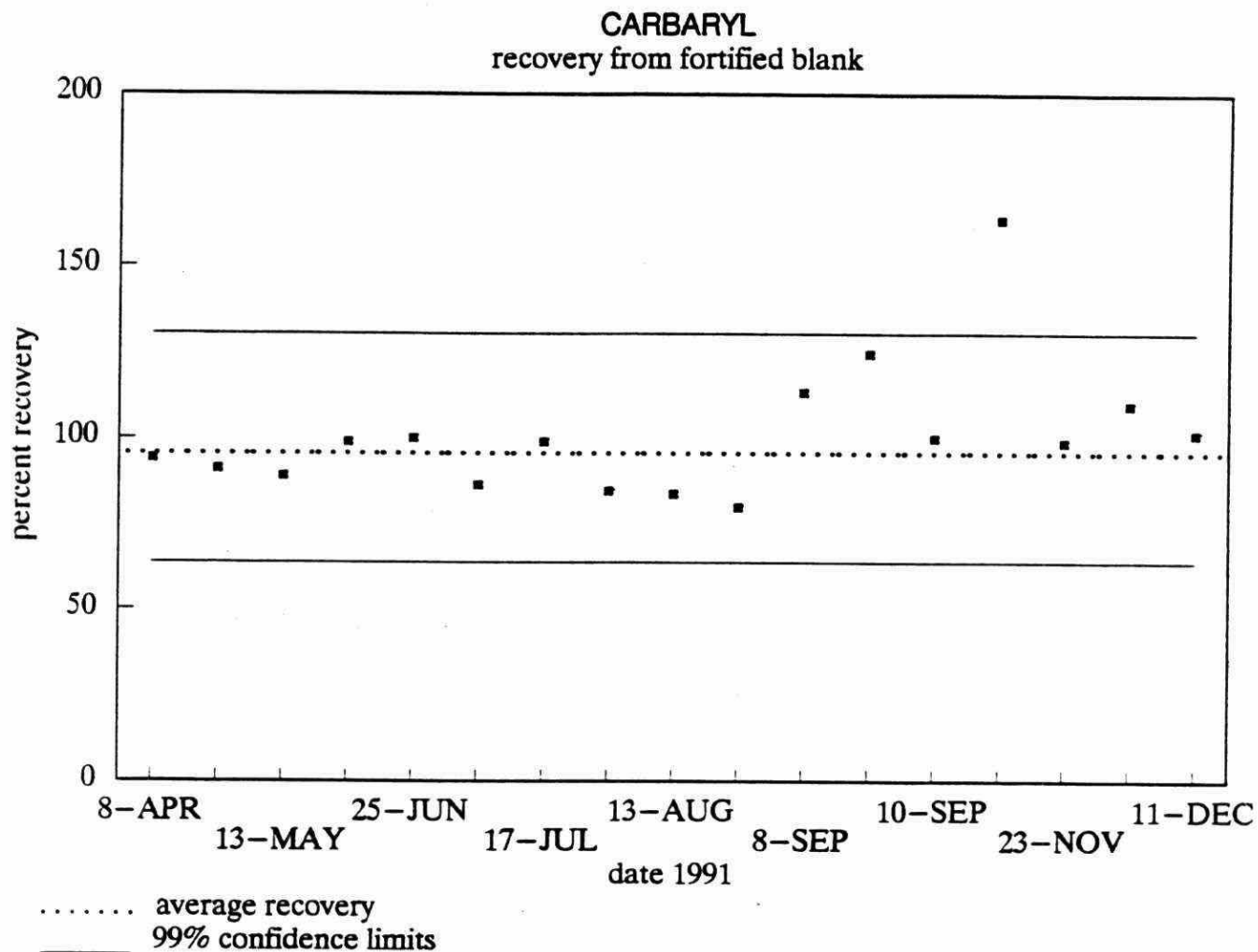
Control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained for selected target compounds.

REMARKS : During the period starting January 1991 and ending December 1991, a total of 16 method blanks was prepared and tested by the method. For these 16 analyses, no observable responses of any of the target analytes were encountered.

In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

List of Performance Charts : Carbaryl (recovery from fortified blank)
Isopropyl-3-chlorophenyl Carbamate (recovery from fortified blank)
Butylate (recovery from fortified blank)

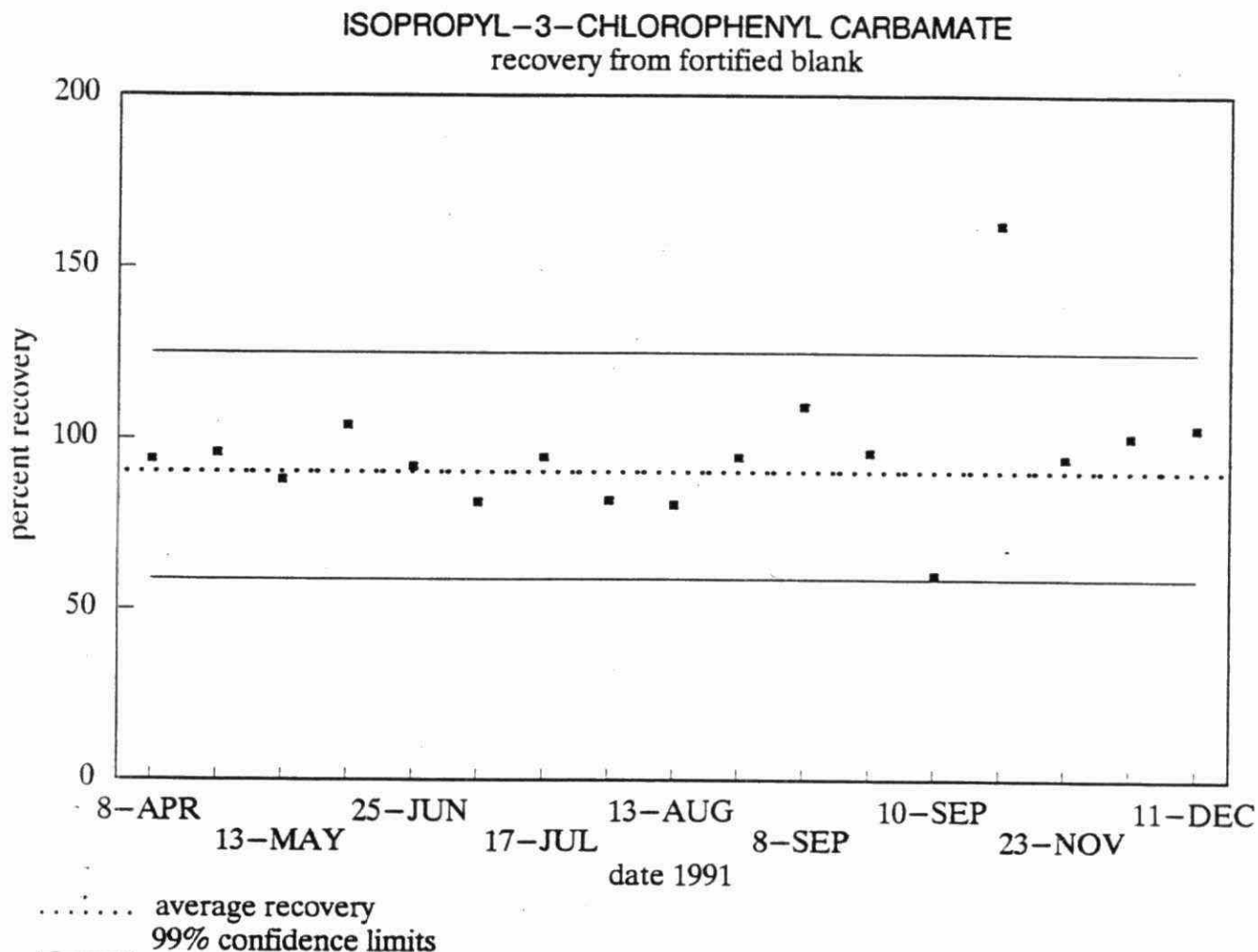
List of Performance Tables : Carbaryl
Isopropyl-3-chlorophenyl Carbamate
Butylate



Performance Summary Table

January - December 1991

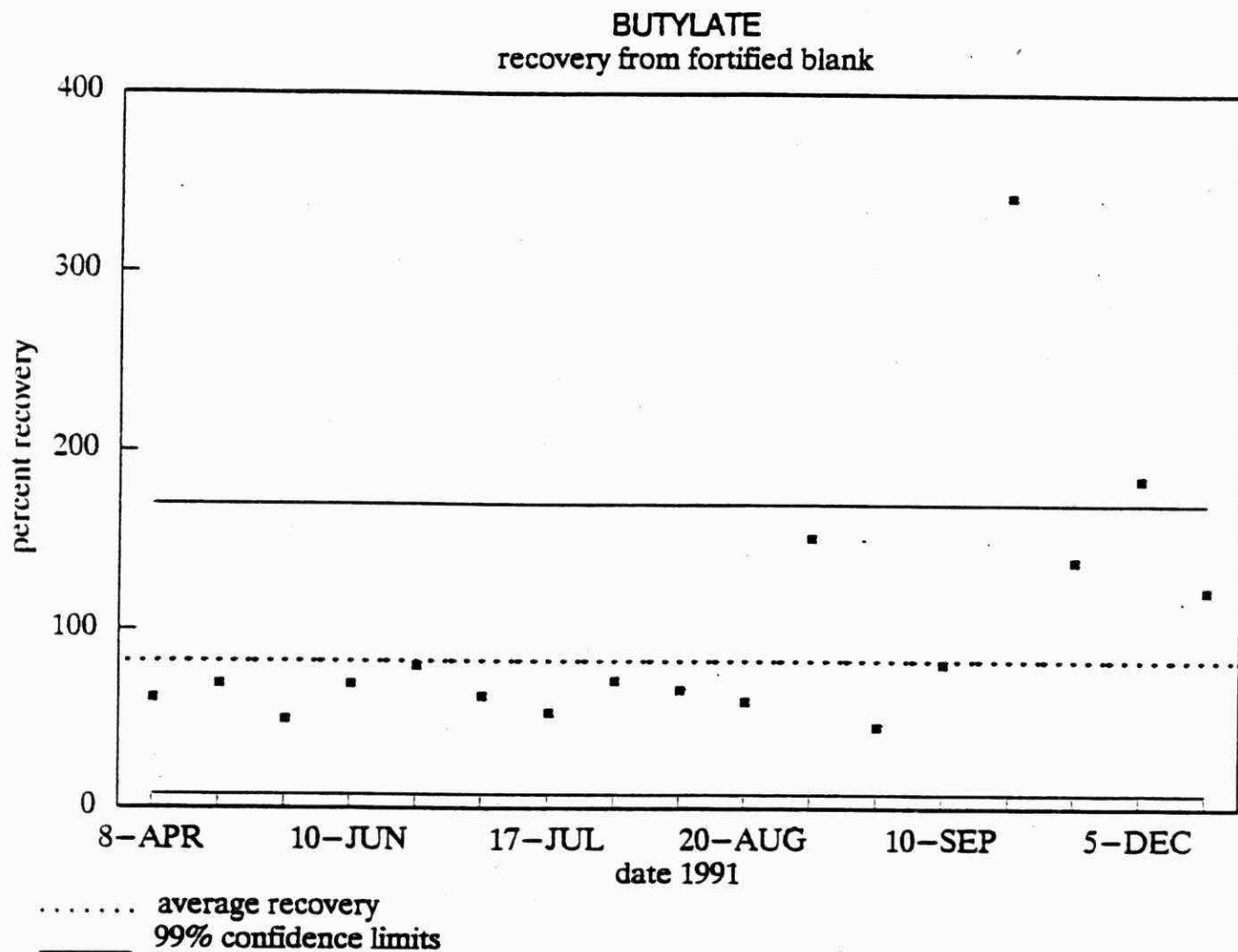
Analyte	carbaryl
True Concentration	10 000 ng/L
Number of Observations	15
Within-run Standard Deviation	not available
Between-run Standard Deviation	11%
Accuracy (% of expected)	97%



Performance Summary Table

January - December 1991

Analyte	CIPC (isopropyl-3-chlorophenyl carbamate)
True Concentration	5 000 ng/L
Number of Observations	15
Within-run Standard Deviation	not available
Between-run Standard Deviation	11%
Accuracy (% of expected)	92%



Performance Summary Table

January - December 1991

Analyte	butylate (Sutan)
True Concentration	5 000 ng/L
Number of Observations	15
Within-run Standard Deviation	not available
Between-run Standard Deviation	31%
Accuracy (% of expected)	79%

METHOD CODE : OWTRI-E3121A.1
METHOD TITLE: The Determination of Triazine Herbicides in Water by GC-TSD
LABORATORY : Organic Water
SUPERVISOR : C.D. Hall

SAMPLE TYPE : surface water, groundwater, finished drinking water

PRINCIPLE OF THE METHOD :

Samples are extracted with solvent, the extract is dried and then evaporated to dryness. The reconstituted extract is examined by gas chromatography using a thermionic specific detector.

PARAMETERS MEASURED :	LIS TEST CODE :	W (ng/L)	T (ng/L)
prometone	P2PROM	50	500
atraton	P2ATRO	50	500
propazine	P2PROP	50	500
atrazine	P2ATRA	50	500
prometryne	P2PROY	50	500
simazine	P2SIM	50	500
ametryne	P2AMET	50	500
sencor	P2SENC	100	1 000
bladex	P2BLAD	100	1 000
metolachlor	P0MET	500	5 000
alachlor	P0LASS	500	5 000

REPORTING FORMAT :

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of W up to maximum of two significant figures.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

Control charts summarizing the response factors used to calibrate instruments and the recoveries from fortified method blanks are maintained for selected target compounds.

REMARKS : In addition to the intra-laboratory method control, the performance of the method was examined through performance audit samples program organized by LSB Quality Management Office.

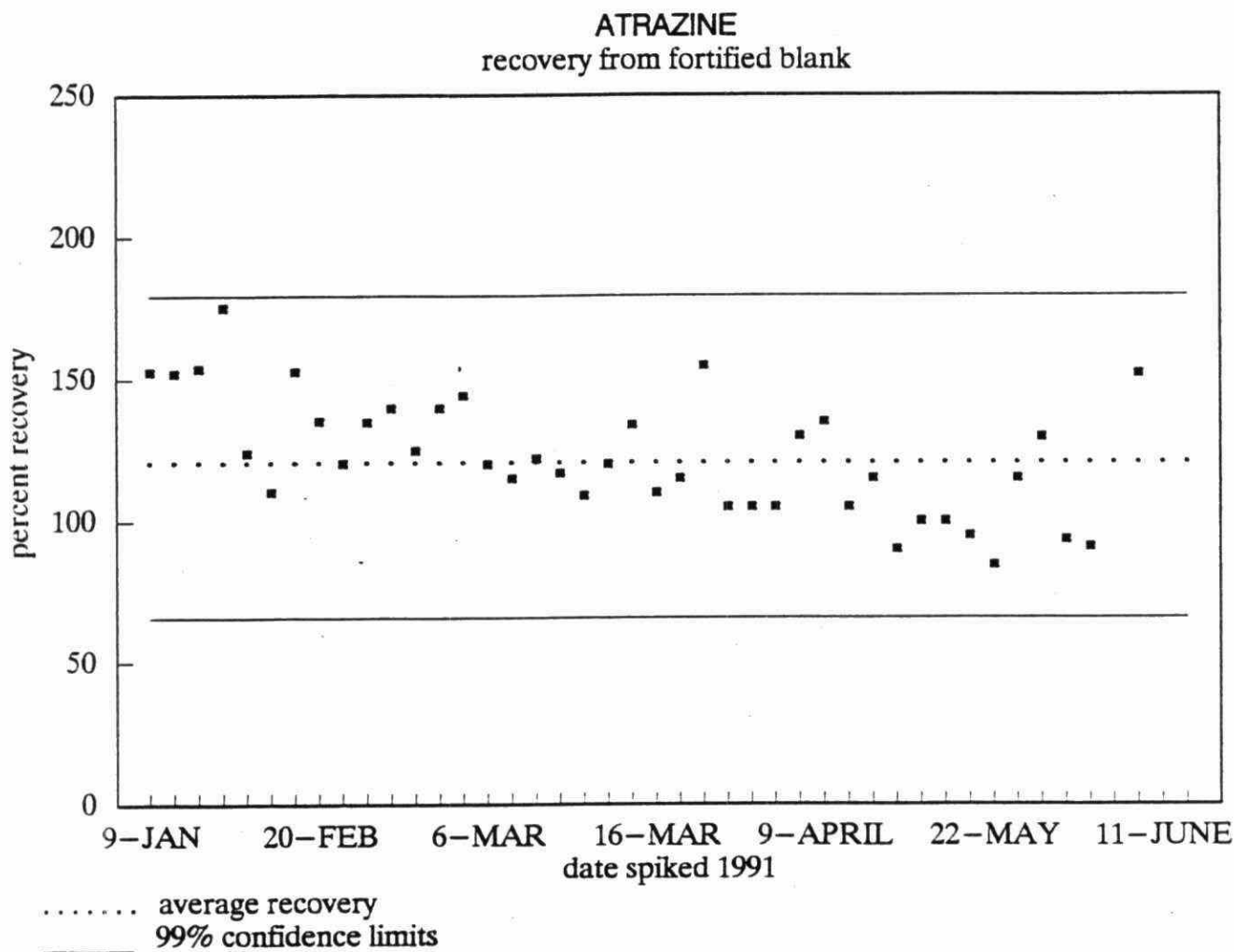
List of Performance Charts : Atrazine (recovery from fortified blank)
Bladex (recovery from fortified blank)
Metolachlor (recovery from fortified blank)

List of Performance Tables : Method Blanks Summary
Atrazine
Bladex
Metolachlor

Method Blanks Summary January 1991 - December 1991

Analyte	Number of Observations	Average Concentration (ng/L)	Standard Deviation (ng/L)
prometone	84	0	0
atraton	84	0	0
propazine	84	0	0
atrazine	84	25 *	51 *
prometryne	84	0	0
simazine	84	0	0
ametryne	84	0	0
sencor	84	0	0
bladex	84	0	0
metolachlor	84	0	0
alachlor	84	0	0

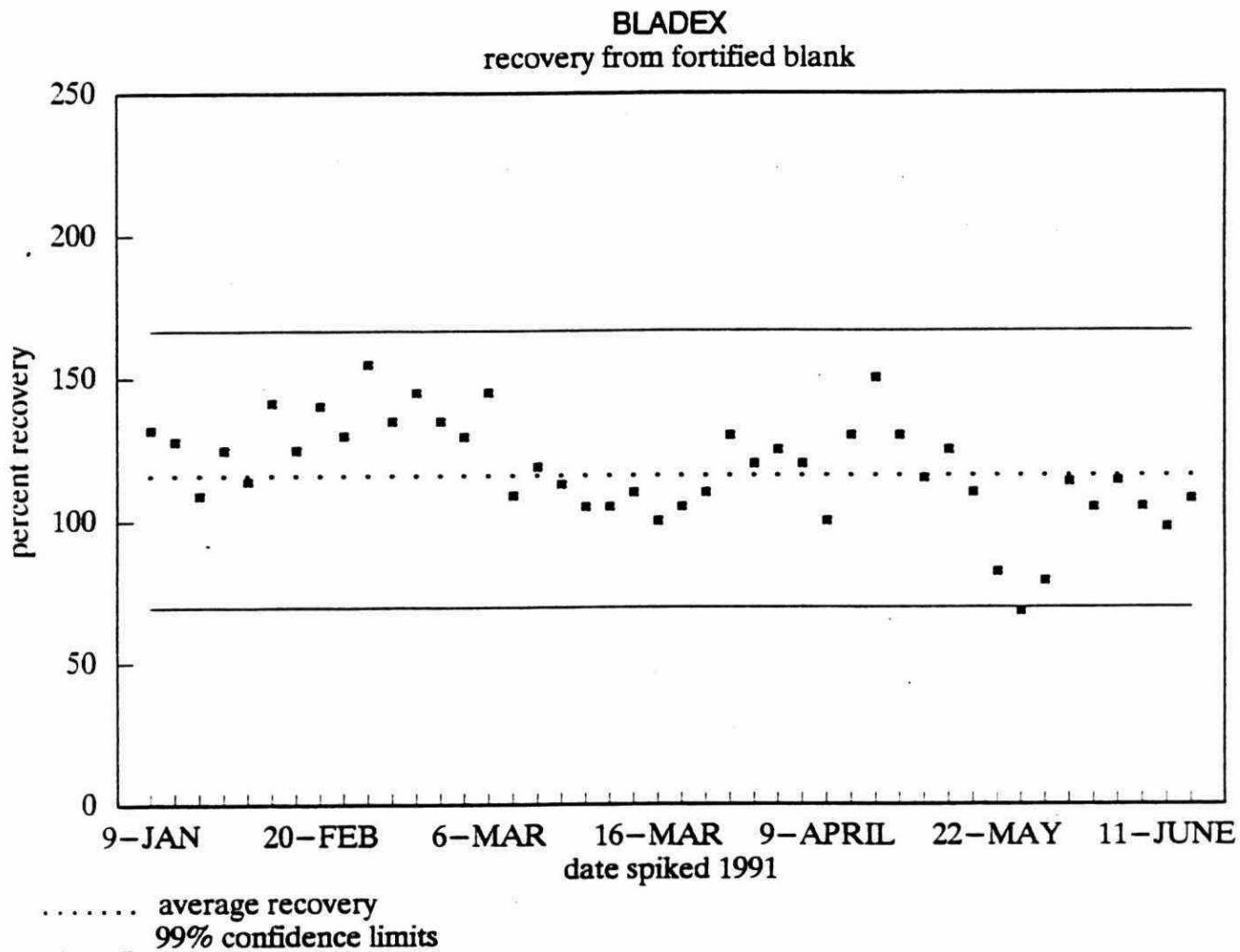
* The trace levels of atrazine were found in method blanks prepared from tap water. In June 1991, Nanopure™ water started to be used for method blanks. No observable concentrations of atrazine were found in any of the total of 38 Nanopure™ water method blanks which were prepared and analyzed in the period starting June 1991 and ending December 1991.



Performance Summary Table

January - December 1991

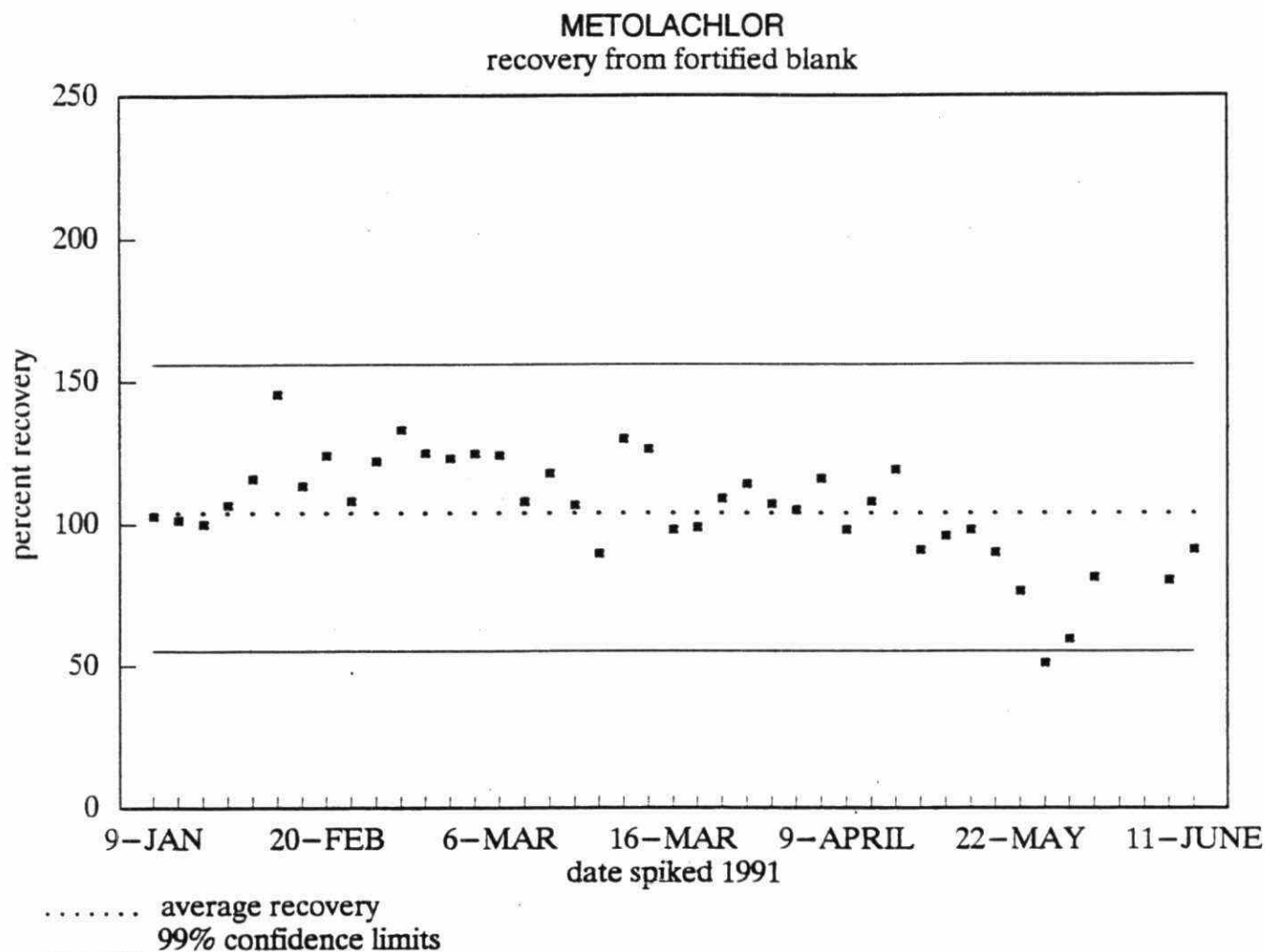
Analyte	atrazine
True Concentration	200 ng/L
Number of Observations	41
Within-run Standard Deviation	not available
Between-run Standard Deviation	21%
Accuracy (% of expected)	122%



Performance Summary Table

January - December 1991

Analyte	bladex
True Concentration	200 ng/L
Number of Observations	44
Within-run Standard Deviation	not available
Between-run Standard Deviation	18%
Accuracy (% of expected)	118%



Performance Summary Table

January - December 1991

Analyte	metolachlor
True Concentration	1 000 ng/L
Number of Observations	41
Within-run Standard Deviation	not available
Between-run Standard Deviation	19%
Accuracy (% of expected)	106%

METHOD CODE : PWAUH-E3230A.1
METHOD TITLE: The Determination of Phenyl Ureas in Water by High Performance Liquid Chromatography

LABORATORY : Organic Water Unit
SUPERVISOR : C.D. Hall

SAMPLE TYPE : surface water, groundwater, finished drinking water

PRINCIPLE OF THE METHOD :

Samples are extracted with an organic solvent; the extract is filtered through granular anhydrous sodium sulphate to remove water and evaporated to dryness by rotary evaporator. The reconstituted extract is examined by high performance liquid chromatography using a variable wavelength ultraviolet detector.

PARAMETERS MEASURED :	LIS TEST CODE :	W (ng/L)	T (ng/L)
linuron	P5LINU	2 000	20 000
monuron	P5MONU	2 000	20 000
diuron	P5DIUR	2 000	20 000
chlortoluron	P5CTOL	2 000	20 000
fluometuron	P5FMET	2 000	20 000
monolinuron	P5MLIN	2 000	20 000
chlorbromuron	P5CBRO	2 000	20 000
metoxuron	P5METX	2 000	20 000
siduron	P5SID	2 000	20 000
difenoxuron	P5DIF	2 000	20 000
neburon	P5NEB	2 000	20 000
paratan	P5PATO	2 000	20 000

REPORTING FORMAT :

Results are reported in parts per trillion (ng/L) rounded off to the closest increment of 100 ng/L and up to maximum of two significant figures.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration (calibration check solution), absence of potential interferences (method blanks), overall method performance (fortified method blanks).

REMARKS : During the period starting January 1991 and ending December 1991, a total of 3 method blanks was prepared and tested by the method. For these 3 analyses, no observable responses of any of the target analytes were encountered.

Since this method is not used on regular basis (less than 50 samples are analyzed per year), no control charts are maintained.

List of Performance Charts : not applicable

List of Performance Tables : Recoveries of Target Analytes from Fortified Method Blanks

Performance Summary Table

Recoveries of PWAUH Target Analytes from Fortified Method Blanks

Analyte	concentration (ng/L)	number of obs.	accuracy (% of expected)	standard deviation (%)
metoxuron	5 000	3	89%	7
monuron	5 000	3	91%	2
chlortoluron	5 000	3	102%	10
fluometuron	5 000	3	105%	12
diuron	5 000	3	106%	10
monolinuron	5 000	3	101%	10
difenoxuron	5 000	3	101%	12
metobromuron	5 000	3	104%	7
siduron	5 000	3	96%	12
linuron	5 000	3	104%	10
chlorbromuron	5 000	3	105%	10
neburon	5 000	3	100%	11

METHOD CODE : PAAFD-E3123A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) in Ambient Air

LABORATORY : Dioxin Unit

SUPERVISOR : E. Reiner

SAMPLE TYPE : ambient air

PRINCIPLE OF THE METHOD :

Samples are collected using a MOE-modified high-volume air sampler with a polyurethane foam (PUF) plug and Teflon-coated glass fiber filter paper. PCDDs and PCDFs are extracted from the PUF and filter paper using a Soxhlet extraction apparatus and toluene. The concentrated extract is processed through a multi-stage chromatographic cleanup procedure to remove the bulk of the sample matrix and potential chemical interferences. After cleanup, the extract is evaporated to dryness.

The reconstituted extract is examined by gas chromatography - triple quadrupole tandem mass spectrometry (GC-MS-MS) or gas chromatography - high resolution mass spectrometry (GC-HRMS).

PARAMETERS MEASURED :

total tetrachlorinated dibenzo-p-dioxins (TCDD)
total pentachlorinated dibenzo-p-dioxins (PCDD)
total hexachlorinated dibenzo-p-dioxins (HxCDD)
total heptachlorinated dibenzo-p-dioxins (HpCDD)
total octachlorinated dibenzo-p-dioxins (OCDD)
total tetrachlorinated dibenzofurans (TCDF)
total pentachlorinated dibenzofurans (PCDF)
total hexachlorinated dibenzofurans (HxCDF)
total heptachlorinated dibenzofurans (HpCDF)
total octachlorinated dibenzofurans (OCDF)

2,3,7,8-tetrachlorodibenzo-p-dioxin
1,2,3,7,8-pentachlorodibenzo-p-dioxin
three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin
2,3,7,8-tetrachlorodibenzofuran
2,3,4,7,8-pentachlorodibenzofuran
1,2,3,7,8-pentachlorodibenzofuran
four 2,3,7,8-substituted hexachlorodibenzofurans

(parameters measured continued)

1,2,3,4,6,7,8-heptachlorodibenzofuran
1,2,3,4,7,8,9-heptachlorodibenzofuran
1,2,3,4,6,7,8,9-octachlorodibenzofuran

REPORTING FORMAT :

Results are reported as pg/m^3 rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific * and range from 0.001 pg/m^3 to 0.01 pg/m^3 .

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, results are reported uncorrected for internal standard recovery.

REMARKS : The performance of the method was examined through CCME Interlaboratory Study PCDD/PCDF in Ambient Air.

Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts : $^{13}\text{C}_{12}$ -Tetrachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Pentachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Hexachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Heptachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Octachlorodibenzo-p-dioxin (recovery of internal standard)

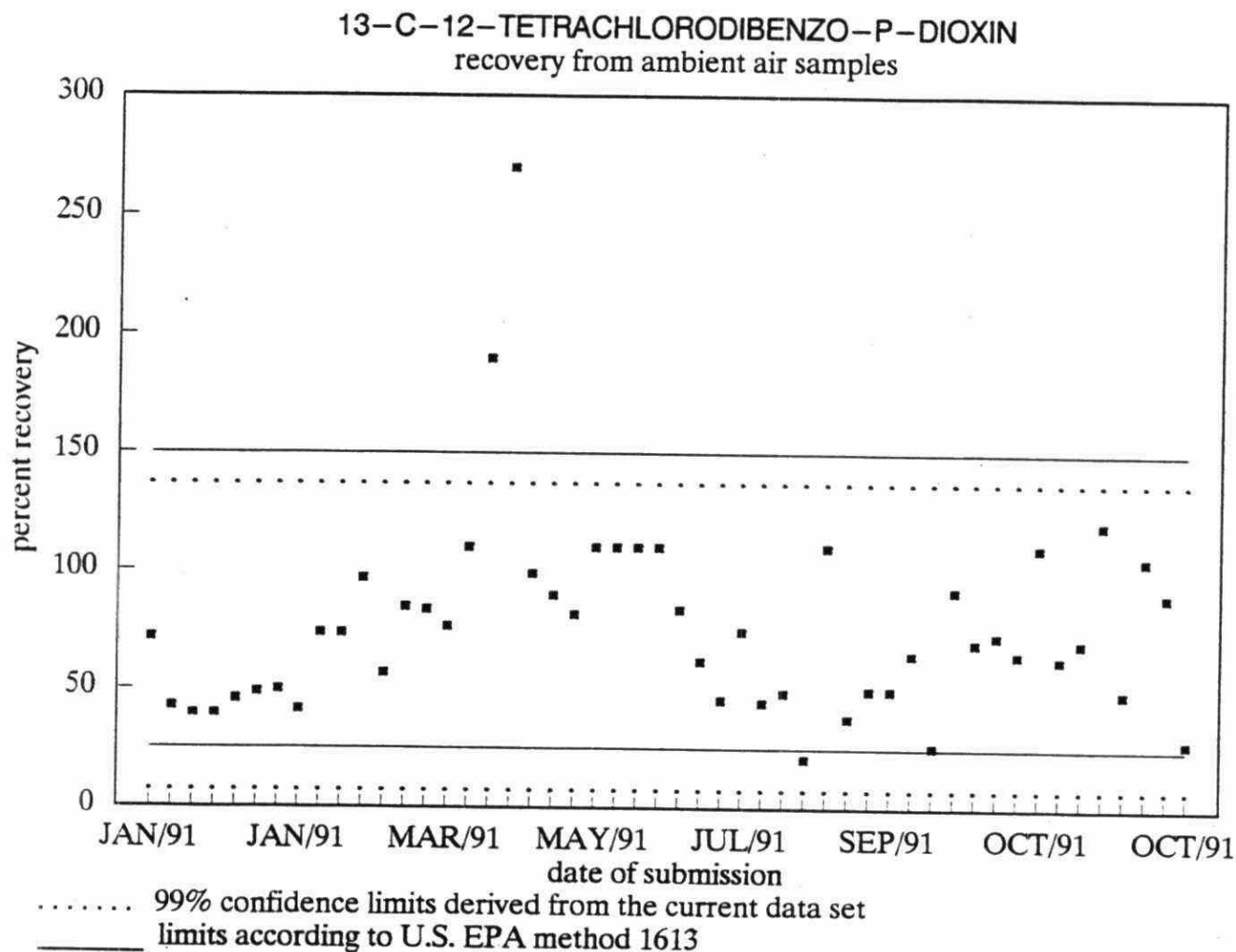
List of Performance Tables : Method Blanks Summary
 $^{13}\text{C}_{12}$ -2,3,7,8-Tetrachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -1,2,3,7,8-Pentachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -Octachlorodibenzo-p-dioxin

* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary

January 1991 - December 1991

Analyte	Number of Observations	Average Concentration ($\times 10^{-3}$ pg/m ³)	Standard Deviation ($\times 10^{-3}$ pg/m ³)
total TCDD	9	0	0
total PCDD	9	0	0
total HxCDD	9	0.9	2.5
total HpCDD	9	0	0
total OCDD	9	7.4	8.8
total TCDF	9	0	0
total PCDF	9	0	0
total HxCDF	9	0	0
total HpCDF	9	0	0
total OCDF	9	0	0

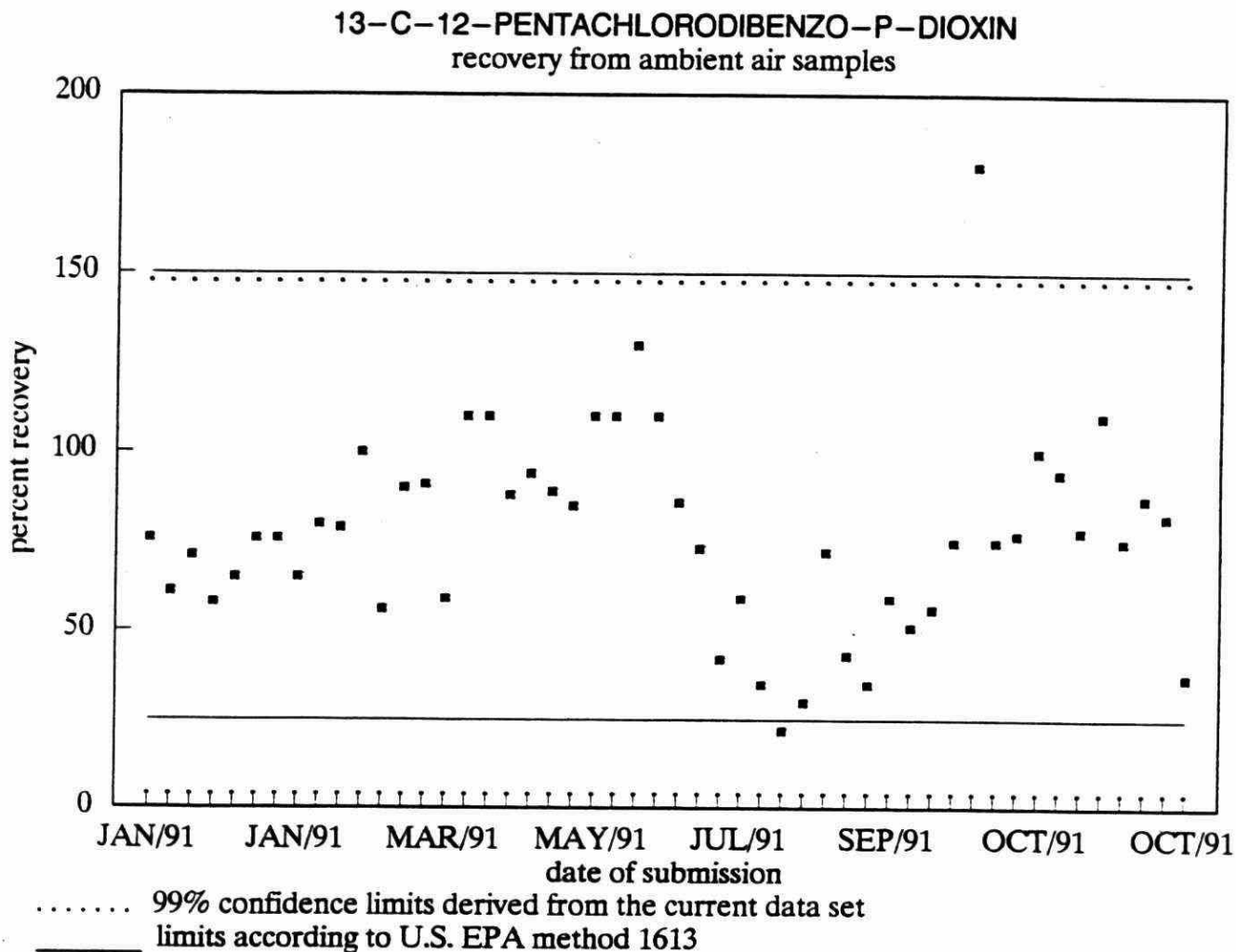


Performance Summary Table

January - December 1991

Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -2,3,7,8-tetrachlorodibenzo-p-dioxin
True Concentration	1 pg/m ³ *
Number of Observations	50
Within-run Rel. Standard Deviation	9% (n=7)
Between-run Standard Deviation	26%
Accuracy (% of expected)	71%

* true concentration relates to the original sample volume of 3 000 m³; see official text of the method for the details on spiking procedure



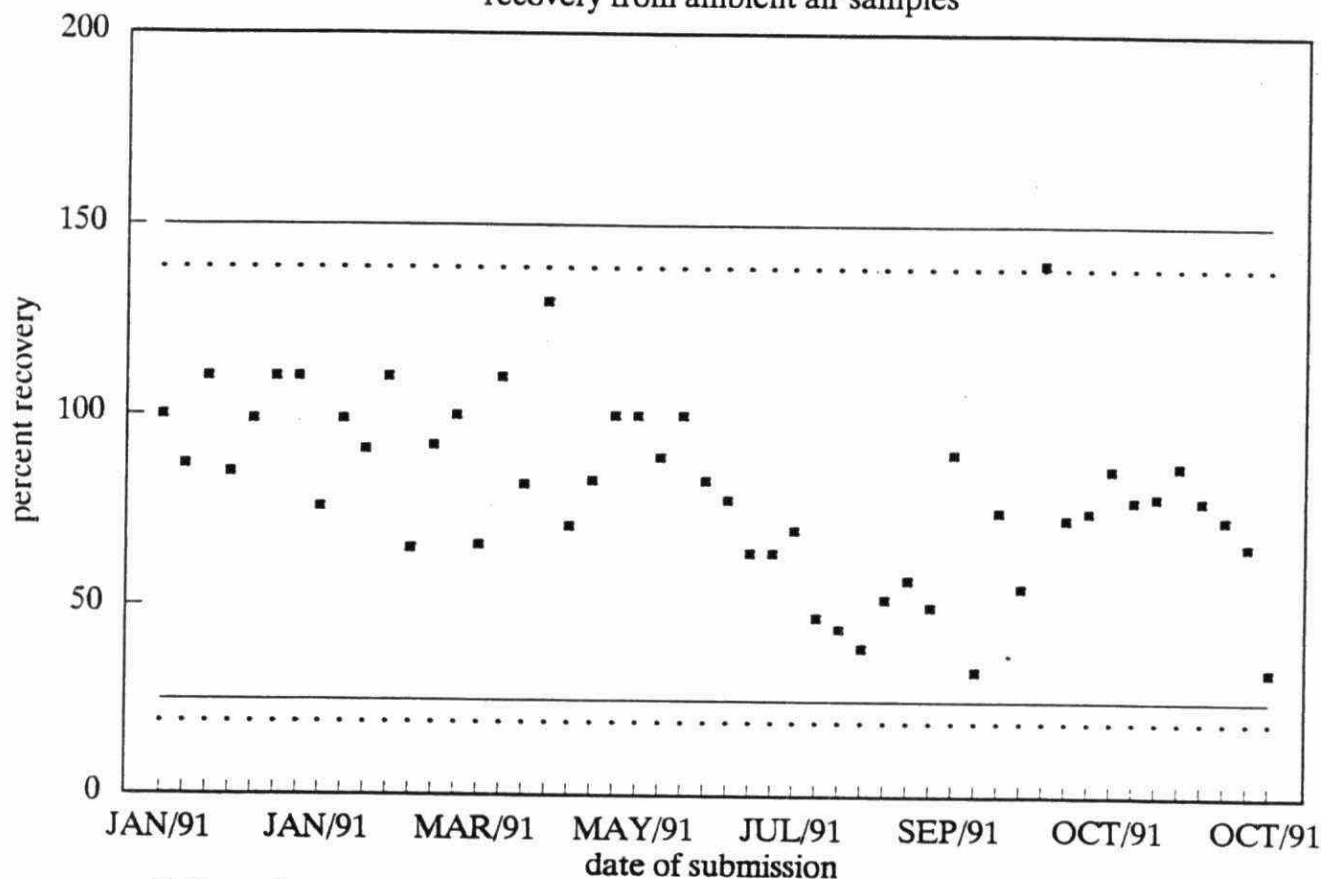
Performance Summary Table

January - December 1991

Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -1,2,3,7,8-pentachlorodibenzo-p-dioxin
True Concentration	1 pg/m ³ *
Number of Observations	50
Within-run Rel. Standard Deviation	19% (n=7)
Between-run Standard Deviation	28%
Accuracy (% of expected)	77%

* true concentration relates to the original sample volume of 3 000 m³; see official text of the method for the details on spiking procedure

¹³C-12-HEXACHLORODIBENZO-P-DIOXIN
recovery from ambient air samples

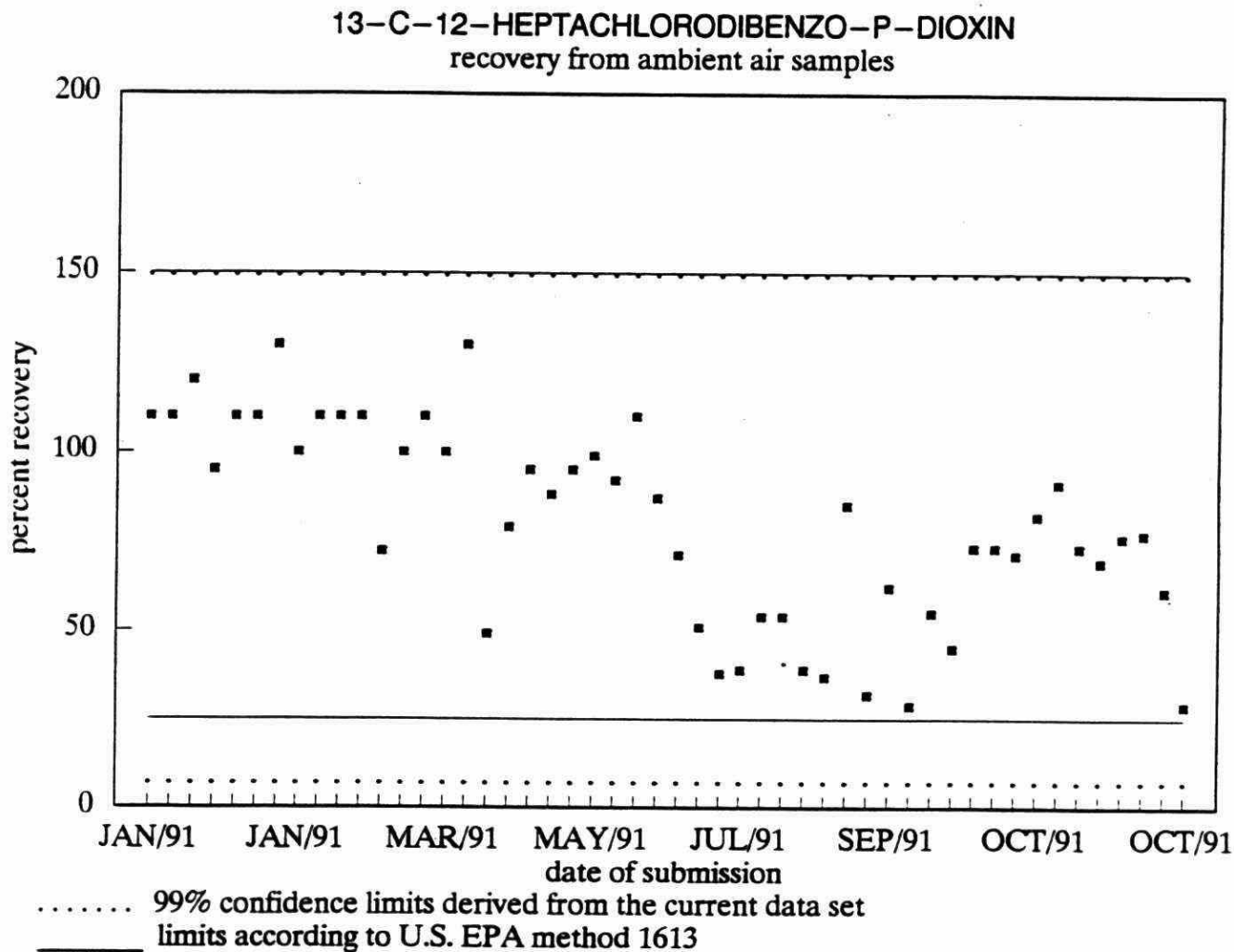


Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin
True Concentration	1 pg/m ³ *
Number of Observations	50
Within-run Rel. Standard Deviation	9% (n=7)
Between-run Standard Deviation	23%
Accuracy (% of expected)	81%

* true concentration relates to the original sample volume of 3 000 m³; see official text of the method for the details on spiking procedure



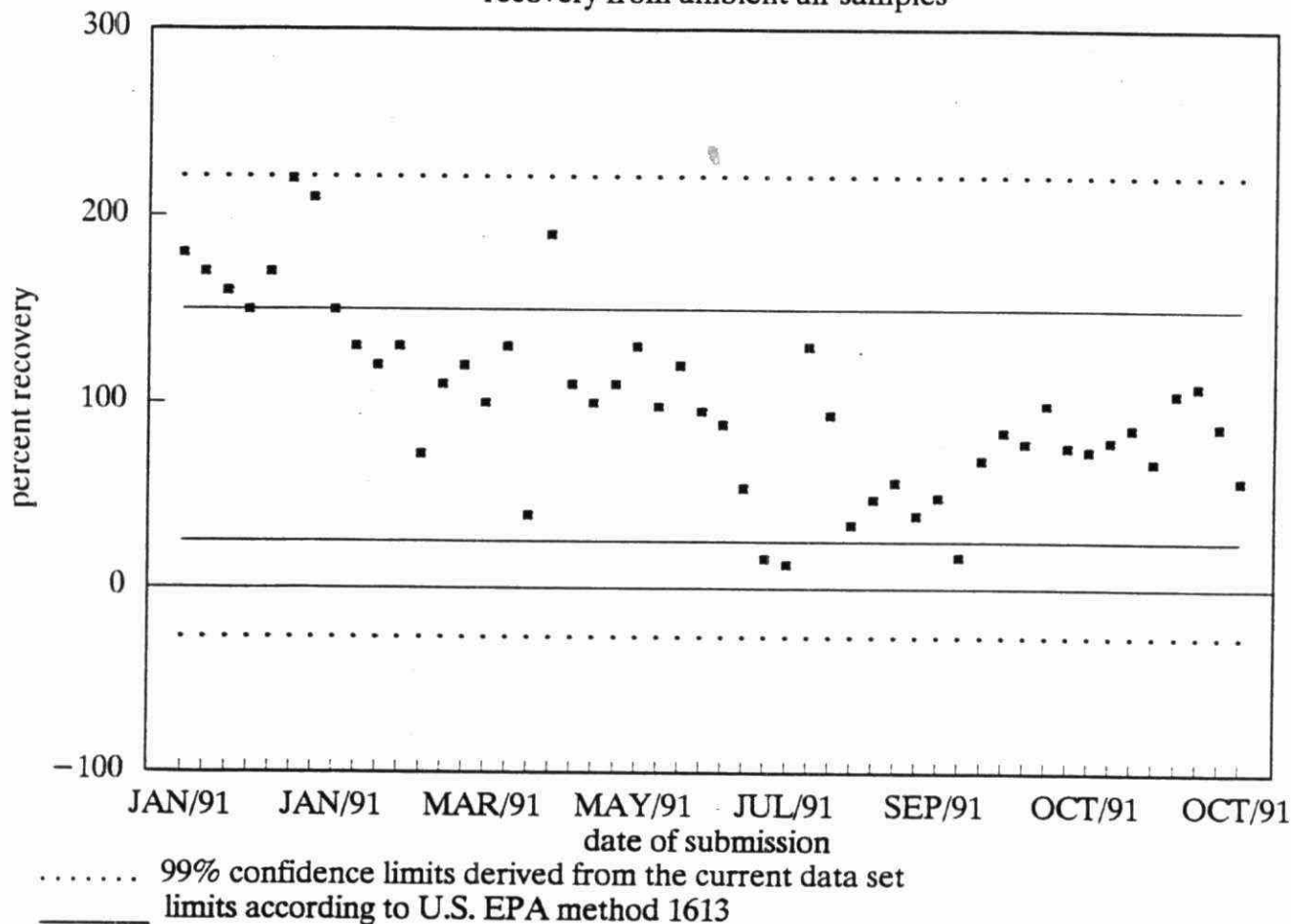
Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
True Concentration	4 pg/m ³ *
Number of Observations	50
Within-run Rel. Standard Deviation	10% (n=7)
Between-run Standard Deviation	28%
Accuracy (% of expected)	80%

* true concentration relates to the original sample volume of 3 000 m³; see official text of the method for the details on spiking procedure

13-C-12-OCTACHLORODIBENZO-P-DIOXIN
recovery from ambient air samples



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -octachlorodibenzo-p-dioxin
True Concentration	3 pg/m ³ *
Number of Observations	50
Within-run Rel. Standard Deviation	18% (n=7)
Between-run Standard Deviation	48%
Accuracy (% of expected)	100%

* true concentration relates to the original sample volume of 3 000 m³; see official text of the method for the details on spiking procedure

METHOD CODE : PFAFD-E3135A.1
METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Fish Tissue

LABORATORY : Dioxin Unit
SUPERVISOR : E. Reiner

SAMPLE TYPE : fish tissue and other biological tissue (clams, shrimps)

PRINCIPLE OF THE METHOD :

Sample is homogenized by mechanical grinding. A homogeneous portion to be analyzed is fortified with isotopically labelled internal standard and is digested overnight with concentrated hydrochloric acid. The resulting solution is extracted with hexane and the extract is passed through a column containing anhydrous sodium sulphate and sulphuric acid-silica packing.

The extract is concentrated and subsequently fractionated using high performance liquid chromatography (HPLC). The reconstituted final extract is analyzed by gas chromatography - mass spectrometry or gas chromatography - triple quadrupole tandem mass spectrometry or gas chromatography - high resolution mass spectrometry.

PARAMETERS MEASURED :

total tetrachlorinated dibenzo-p-dioxins (TCDD)
total pentachlorinated dibenzo-p-dioxins (PCDD)
total hexachlorinated dibenzo-p-dioxins (HxCDD)
total heptachlorinated dibenzo-p-dioxins (HpCDD)
total octachlorinated dibenzo-p-dioxins (OCDD)
total tetrachlorinated dibenzofurans (TCDF)
total pentachlorinated dibenzofurans (PCDF)
total hexachlorinated dibenzofurans (HxCDF)
total heptachlorinated dibenzofurans (HpCDF)
total octachlorinated dibenzofurans (OCDF)

2,3,7,8-tetrachlorodibenzo-p-dioxin
1,2,3,7,8-pentachlorodibenzo-p-dioxin
three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin
2,3,7,8-tetrachlorodibenzofuran
2,3,4,7,8-pentachlorodibenzofuran
1,2,3,7,8-pentachlorodibenzofuran
four 2,3,7,8-substituted hexachlorodibenzofurans
1,2,3,4,6,7,8-heptachlorodibenzofuran

(parameters measured continued)

1,2,3,4,7,8,9-heptachlorodibenzofuran
1,2,3,4,6,7,8,9-octachlorodibenzofuran

REPORTING FORMAT :

Results are reported as ppt (picograms of CDD/CDF per gram of wet fish tissue) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific * and range from 1 pg/g to 10 pg/g.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the results are reported uncorrected for internal standard recovery.

REMARKS : Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts : ¹³C₁₂-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Pentachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Hexachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Heptachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Octachlorodibenzo-p-dioxin (recovery of internal standard)

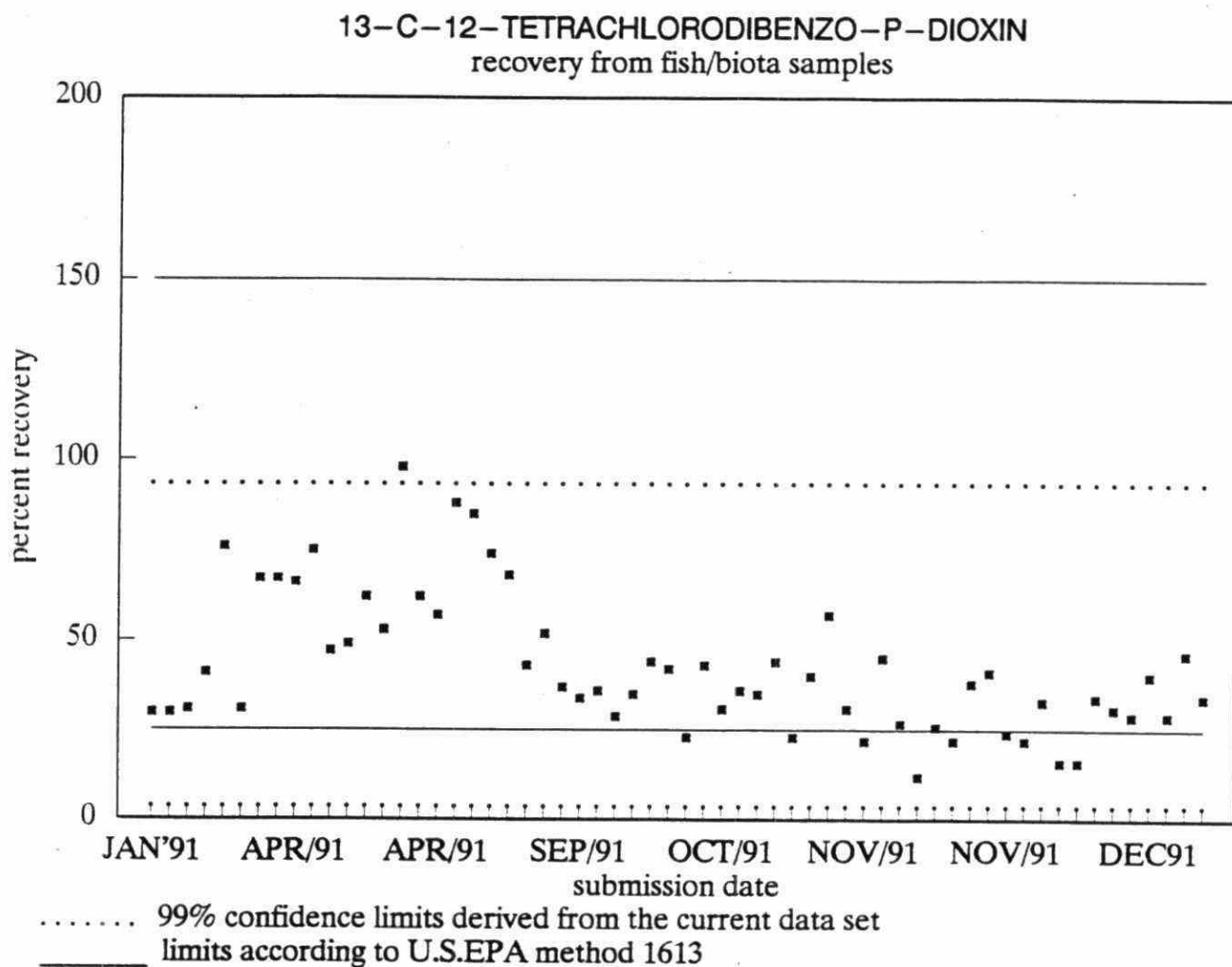
List of Performance Tables : Method Blanks Summary
¹³C₁₂-2,3,7,8-Tetrachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,7,8-Pentachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin
¹³C₁₂-Octachlorodibenzo-p-dioxin

* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary

January 1991 - December 1991

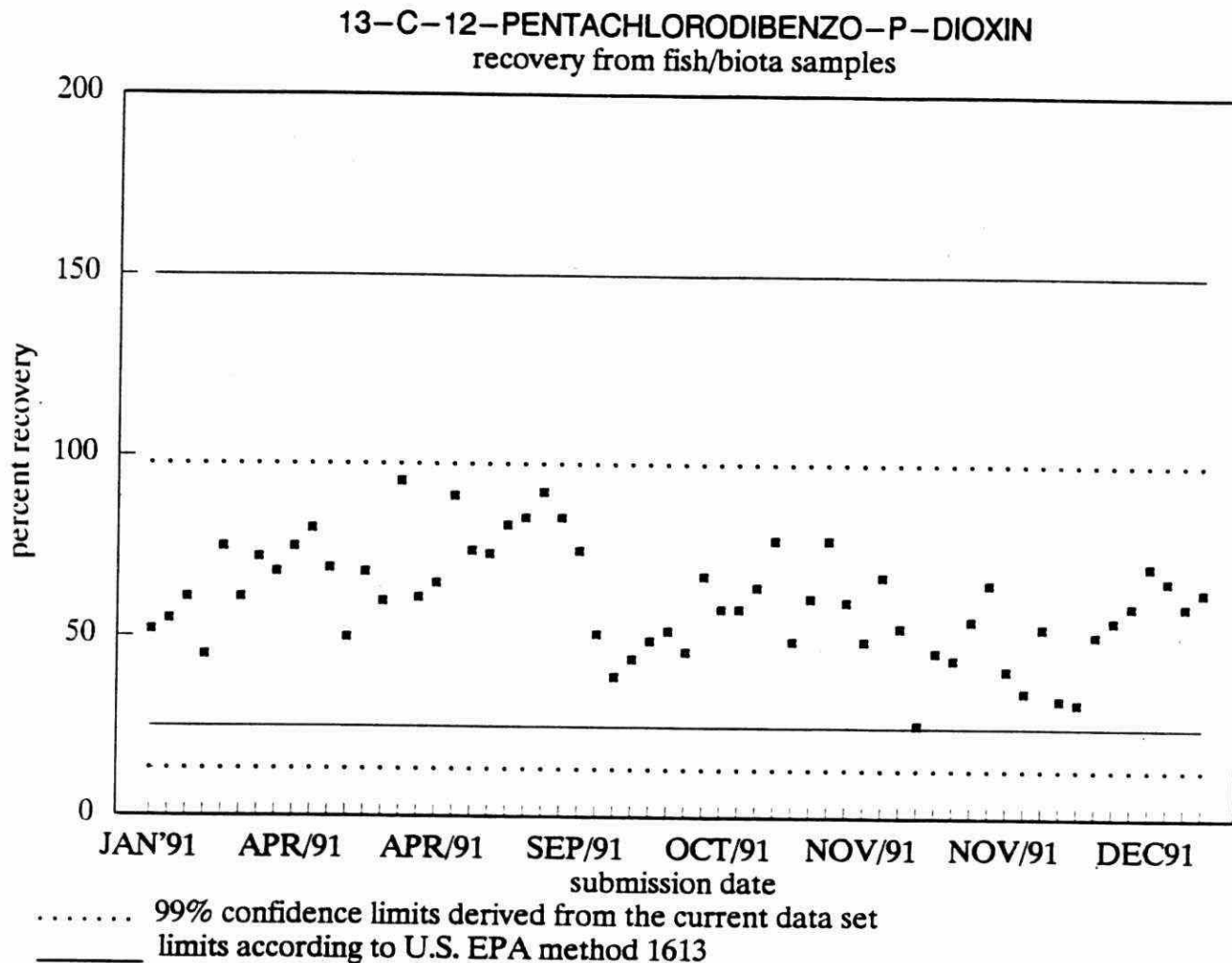
Analyte	Number of Observations	Average Concentration (pg/g)	Standard Deviation (pg/g)
total TCDD	6	0.33	0.47
total PCDD	6	0.17	0.37
total HxCDD	6	0	0
total HpCDD	6	0.17	0.37
total OCDD	6	0.33	0.47
total TCDF	6	0.17	0.37
total PCDF	6	0.17	0.37
total HxCDF	6	0	0
total HpCDF	6	0	0
total OCDF	6	0.17	0.37



Performance Summary Table

January - December 1991

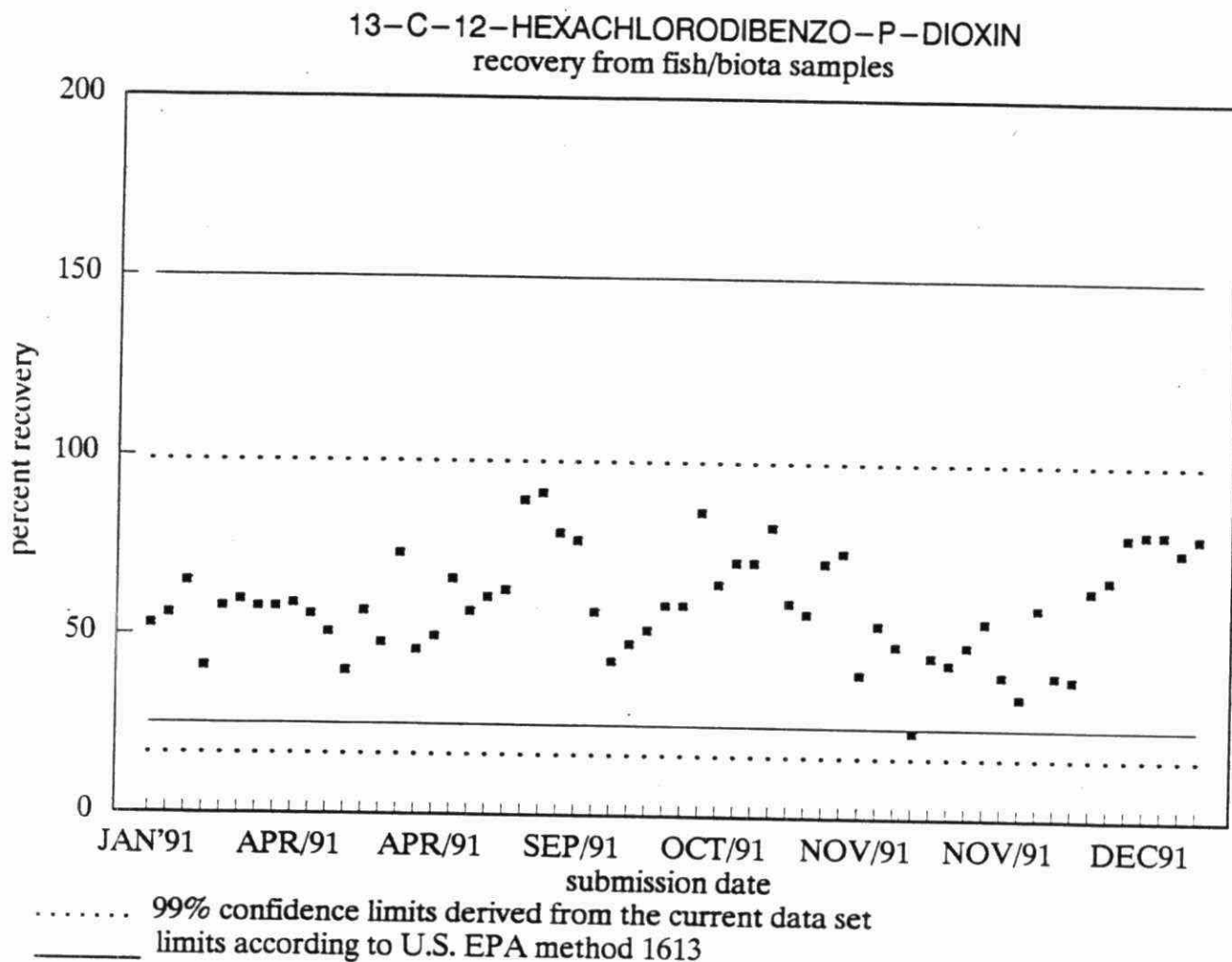
Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -2,3,7,8-tetrachlorodibenzo-p-dioxin
True Concentration	200 pg/g
Number of Observations	60
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	19%
Accuracy (% of expected)	43%



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -1,2,3,7,8-pentachlorodibenzo-p-dioxin
True Concentration	200 pg/g
Number of Observations	60
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	15%
Accuracy (% of expected)	61%

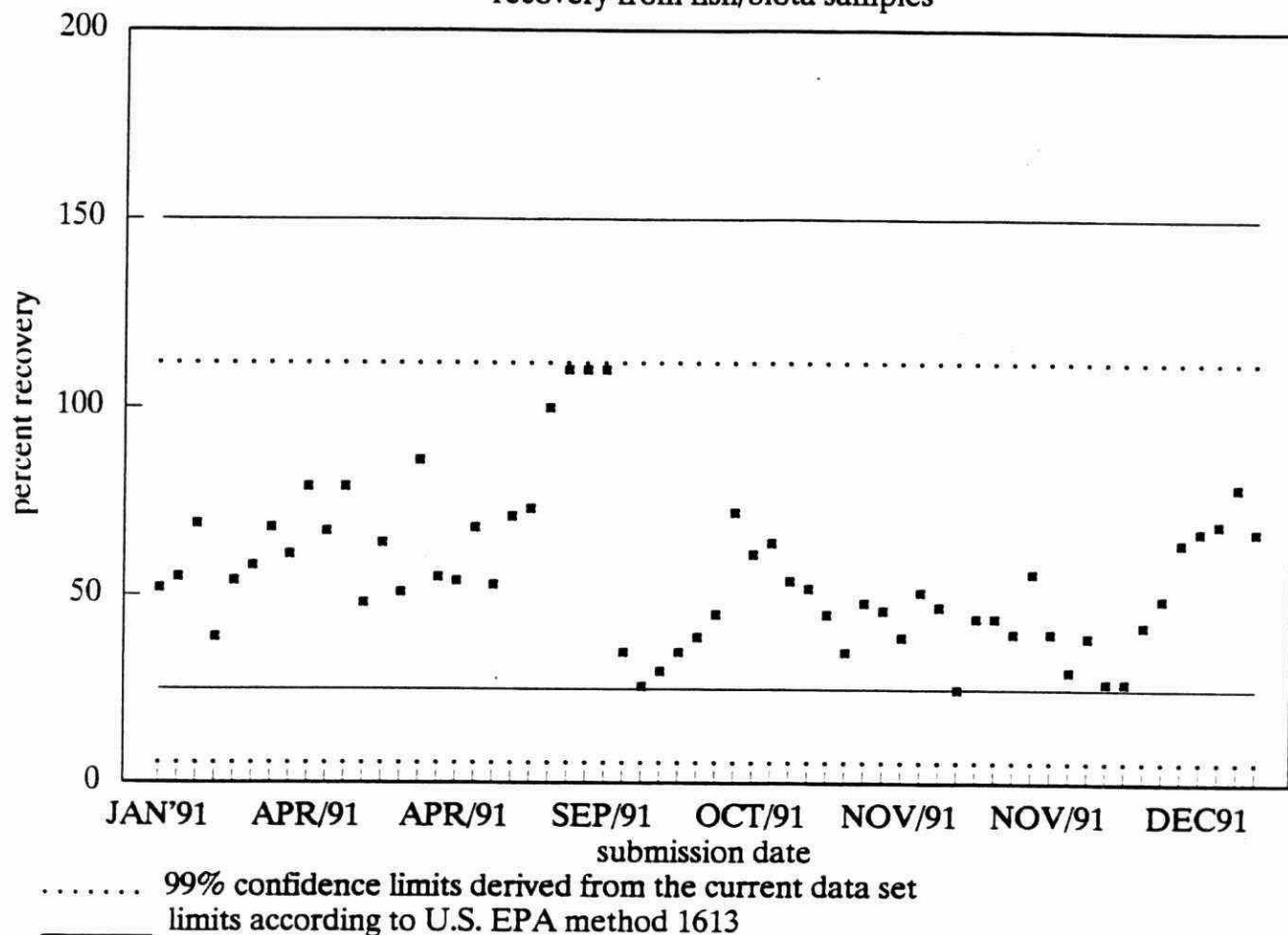


Performance Summary Table

January - December 1991

Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin
True Concentration	170 pg/g
Number of Observations	60
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	20%
Accuracy (% of expected)	59%

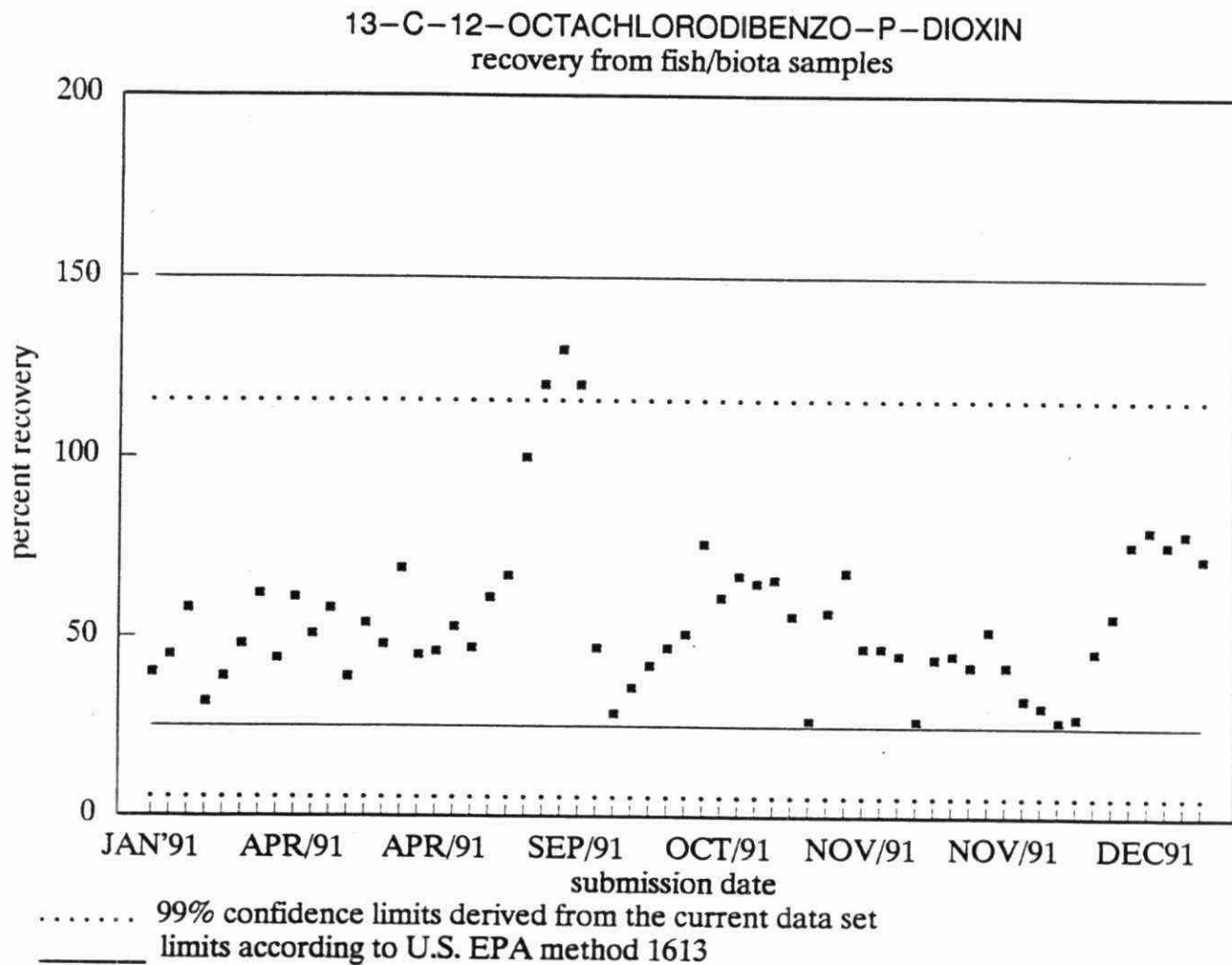
¹³C-12-HEPTACHLORODIBENZO-P-DIOXIN
recovery from fish/biota samples



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
True Concentration	600 pg/g
Number of Observations	60
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	14%
Accuracy (% of expected)	56%



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -octachlorodibenzo-p-dioxin
True Concentration	400 pg/g
Number of Observations	60
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	22%
Accuracy (% of expected)	56%

METHOD CODE : PSAFD-E3152A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins (PCDD) and Polychlorinated Dibenzofurans (PCDF) in Soil and Sediment

LABORATORY : Dioxin Unit

SUPERVISOR : E. Reiner

SAMPLE TYPE : soil and sediment

PRINCIPLE OF THE METHOD :

Samples are dried, ground and homogenized. PCDD and PCDF are extracted from soil/sediment using a Soxhlet extraction apparatus and toluene. The concentrated extract is processed through a multi-stage chromatographic cleanup procedure to remove bulk of the sample matrix and potential chemical interferences.

The reconstituted final extract is analyzed by gas chromatography - mass spectrometry or gas chromatography - triple quadrupole tandem mass spectrometry or gas chromatography - high resolution mass spectrometry.

PARAMETERS MEASURED :

- total tetrachlorinated dibenzo-p-dioxins (TCDD)
- total pentachlorinated dibenzo-p-dioxins (PCDD)
- total hexachlorinated dibenzo-p-dioxins (HxCDD)
- total heptachlorinated dibenzo-p-dioxins (HpCDD)
- total octachlorinated dibenzo-p-dioxins (OCDD)
- total tetrachlorinated dibenzofurans (TCDF)
- total pentachlorinated dibenzofurans (PCDF)
- total hexachlorinated dibenzofurans (HxCDF)
- total heptachlorinated dibenzofurans (HpCDF)
- total octachlorinated dibenzofurans (OCDF)

- 2,3,7,8-tetrachlorodibenzo-p-dioxin
- 1,2,3,7,8-pentachlorodibenzo-p-dioxin
- three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins
- 1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
- 1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin
- 2,3,7,8-tetrachlorodibenzofuran
- 2,3,4,7,8-pentachlorodibenzofuran
- 1,2,3,7,8-pentachlorodibenzofuran
- four 2,3,7,8-substituted hexachlorodibenzofurans
- 1,2,3,4,6,7,8-heptachlorodibenzofuran
- 1,2,3,4,7,8,9-heptachlorodibenzofuran
- 1,2,3,4,6,7,8,9-octachlorodibenzofuran

REPORTING FORMAT :

Results are reported as ppt (picograms of CDD/CDF per gram of soil) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific * and range from 1 pg/g to 10 pg/g.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the results are reported uncorrected for internal standard recovery.

REMARKS : Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts : $^{13}\text{C}_{12}$ -Tetrachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Pentachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Hexachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Heptachlorodibenzo-p-dioxin (recovery of internal standard)
 $^{13}\text{C}_{12}$ -Octachlorodibenzo-p-dioxin (recovery of internal standard)

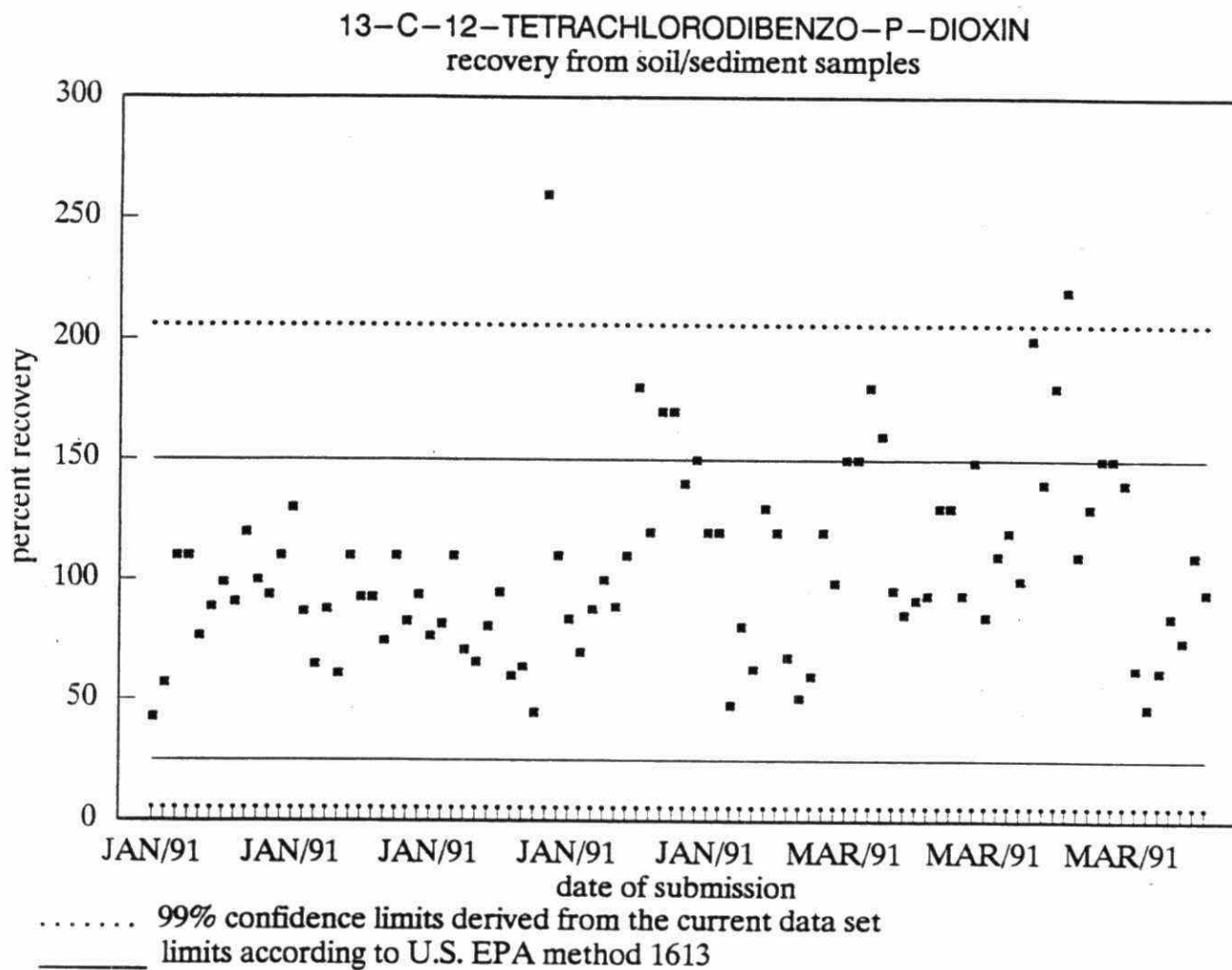
List of Performance Tables : Method Blanks Summary
 $^{13}\text{C}_{12}$ -2,3,7,8-Tetrachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -1,2,3,7,8-Pentachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin
 $^{13}\text{C}_{12}$ -Octachlorodibenzo-p-dioxin

* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary

January 1991 - December 1991

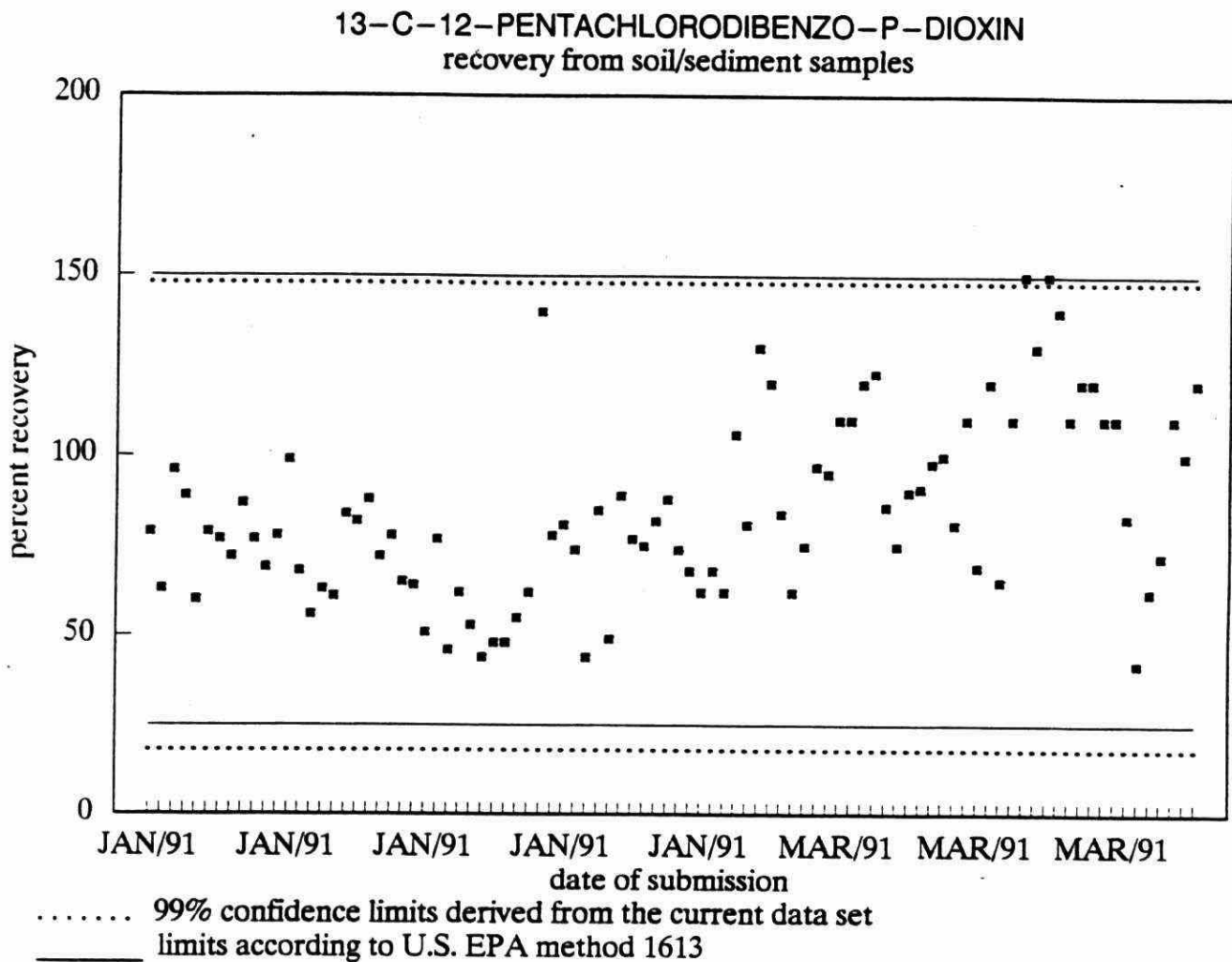
Analyte	Number of Observations	Average Concentration (pg/g)	Standard Deviation (pg/g)
total TCDD	22	0	0
total PCDD	22	0.27	0.69
total HxCDD	22	0.27	1.3
total HpCDD	22	0.09	0.42
total OCDD	22	0.36	0.48
total TCDF	22	0.05	0.21
total PCDF	22	0.05	0.21
total HxCDF	22	0.14	0.34
total HpCDF	22	0.27	0.54
total OCDF	22	0.18	0.39



Performance Summary Table

January - December 1991

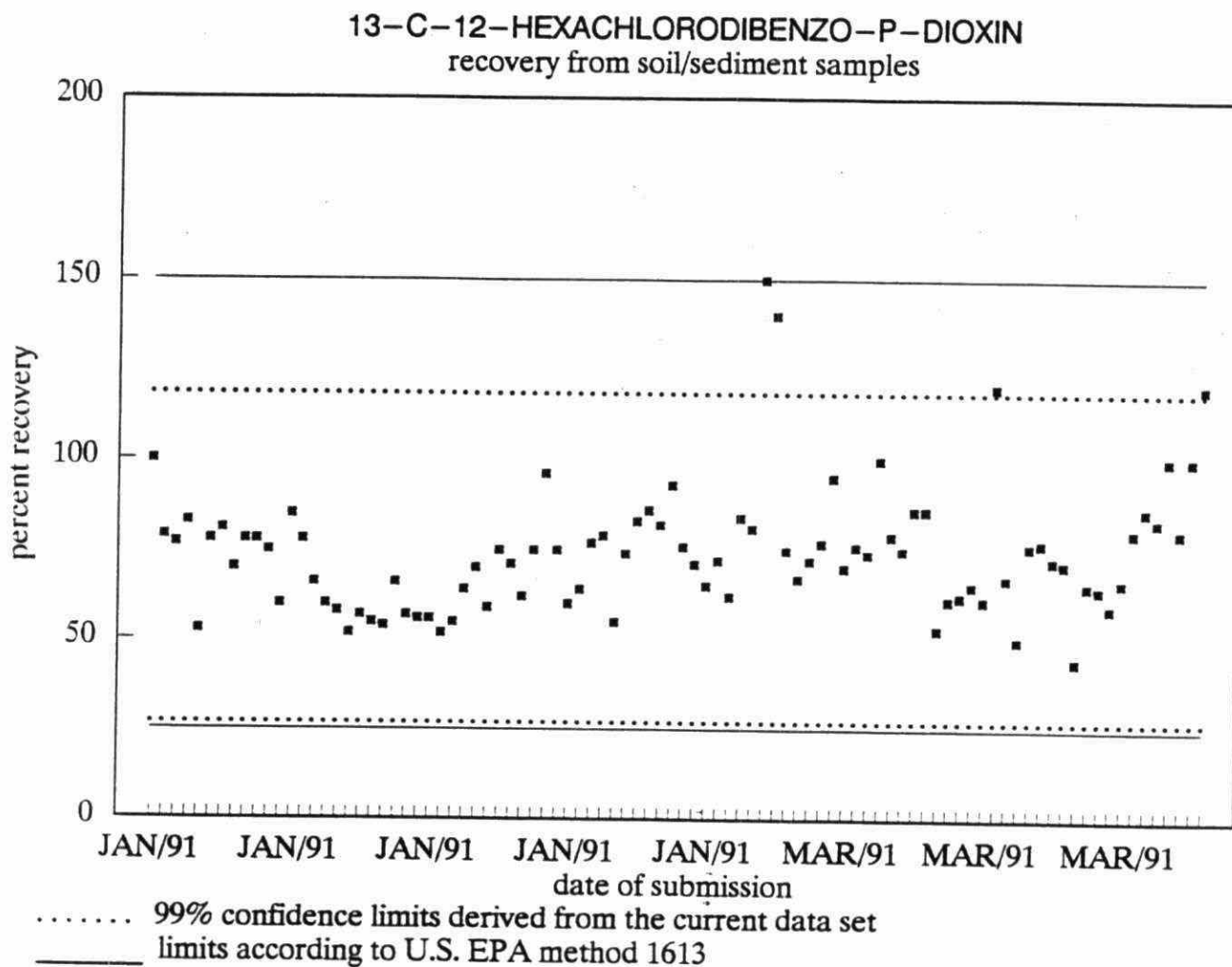
Analyte (Internal Standard)	¹³ C ₁₂ -2,3,7,8-tetrachlorodibenzo-p-dioxin
True Concentration	400 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	17% (n=7)
Between-run Standard Deviation	39%
Accuracy (% of expected)	106%



Performance Summary Table

January - December 1991

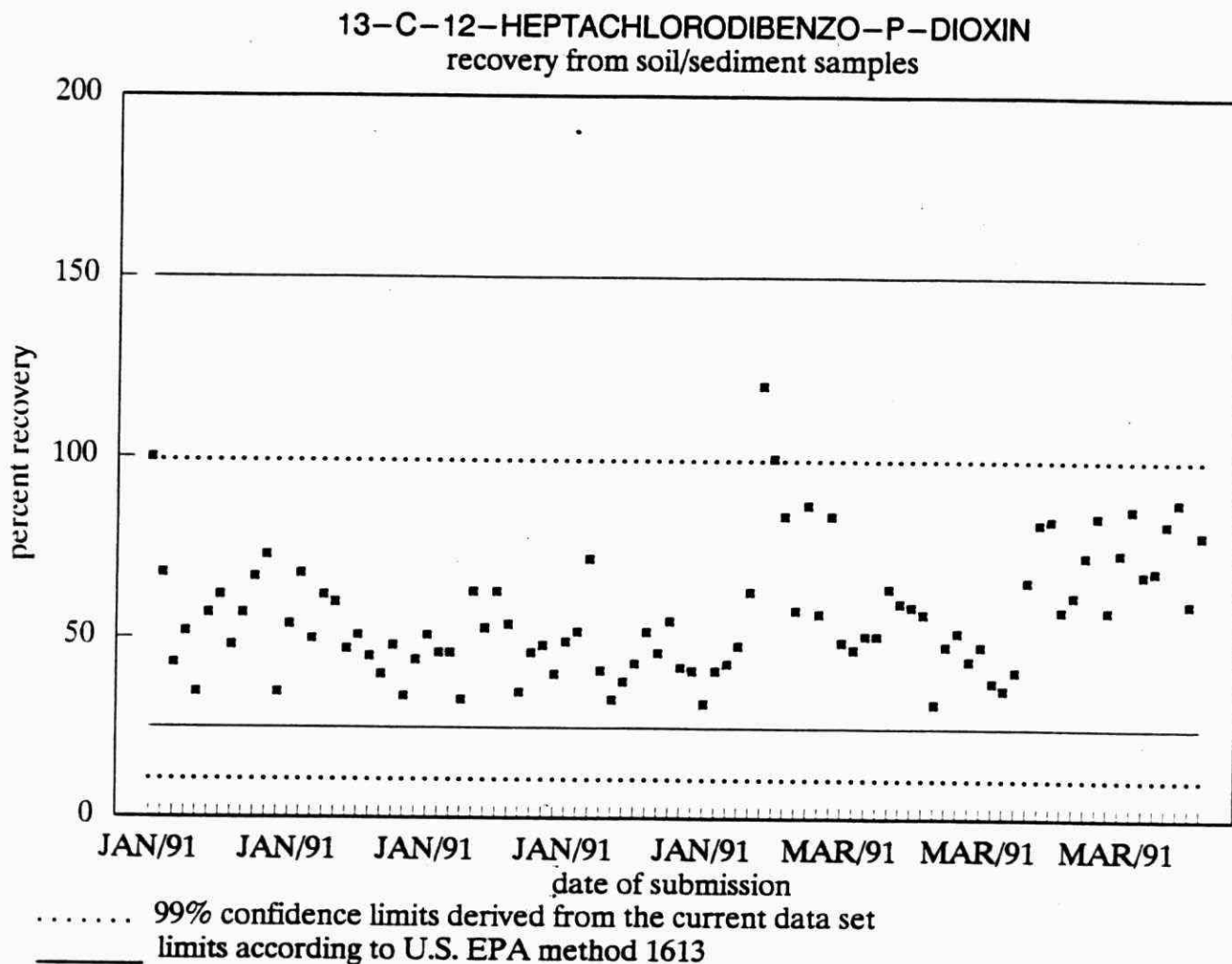
Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,7,8-pentachlorodibenzo-p-dioxin
True Concentration	400 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	11% (n=7)
Between-run Standard Deviation	25%
Accuracy (% of expected)	85%



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin
True Concentration	350 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	3% (n=7)
Between-run Standard Deviation	18%
Accuracy (% of expected)	72%

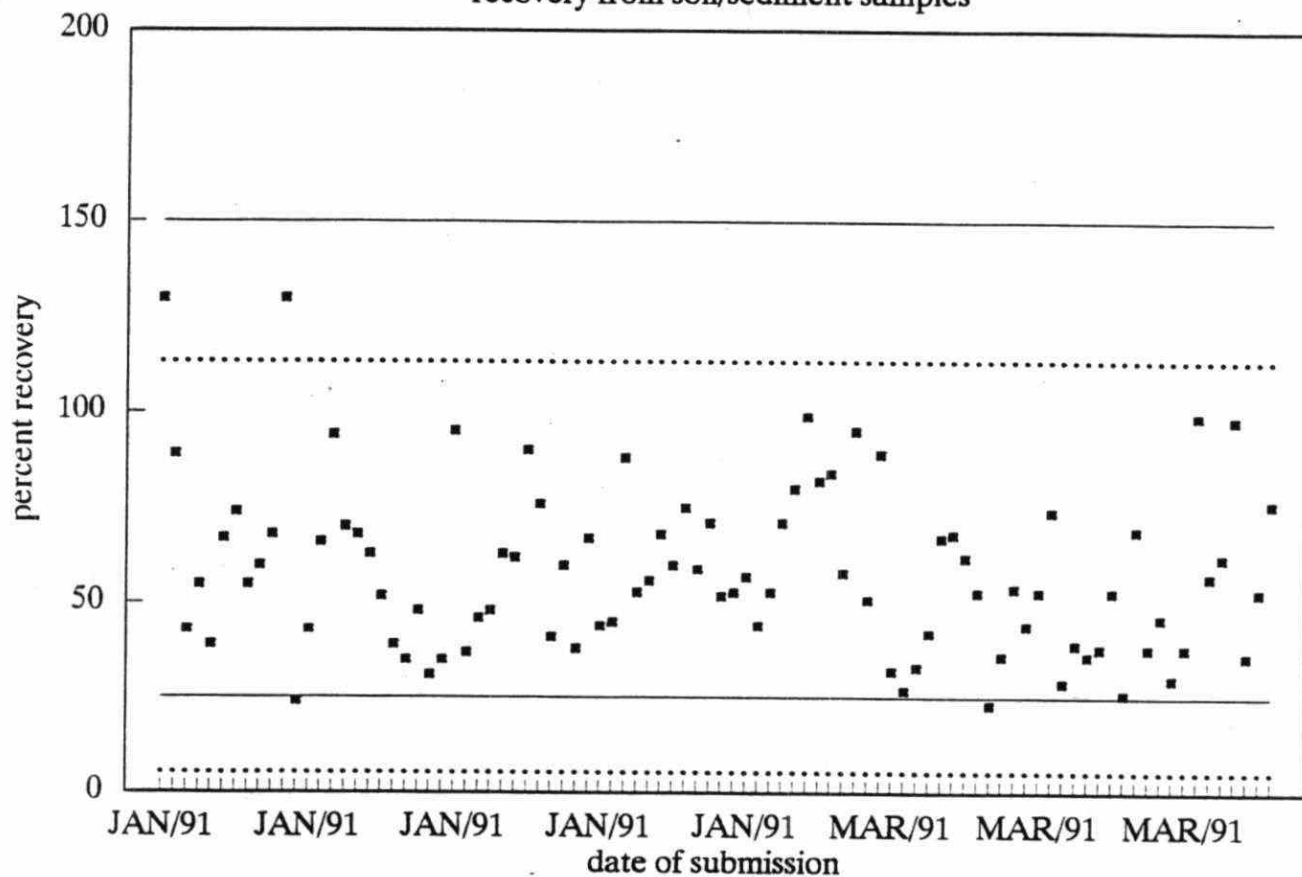


Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
True Concentration	1 200 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	17% (n=7)
Between-run Standard Deviation	17%
Accuracy (% of expected)	57%

13-C-12-OCTACHLORODIBENZO-P-DIOXIN
recovery from soil/sediment samples



..... 99% confidence limits derived from the current data set
 _____ limits according to U.S. EPA method 1613

Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -octachlorodibenzo-p-dioxin
True Concentration	800 pg/g
Number of Observations	92
Within-run Rel. Standard Deviation	6% (n=7)
Between-run Standard Deviation	22%
Accuracy (% of expected)	59%

METHOD CODE : PWAFFD-E3163A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Drinking Water by GC-MS

LABORATORY : Dioxin Unit

SUPERVISOR : E. Reiner

SAMPLE TYPE : raw or finished drinking water

PRINCIPLE OF THE METHOD :

Target analyte(s) are extracted from samples with an organic solvent. The extract is then cleaned of potential chemical interferences by two-stage column chromatography. Cleaned extract is evaporated to dryness. After reconstitution, the extract is examined by gas chromatography - mass spectrometry.

If the extract contains chemical interferences that prevent the quantification of target analytes, it is further fractionated using high performance liquid chromatography and then re-analyzed by GC-MS.

PARAMETERS MEASURED :

total tetrachlorinated dibenzo-p-dioxins
total pentachlorinated dibenzo-p-dioxins
total hexachlorinated dibenzo-p-dioxins
total heptachlorinated dibenzo-p-dioxins
total octachlorinated dibenzo-p-dioxins
total tetrachlorinated dibenzofurans
total pentachlorinated dibenzofurans
total hexachlorinated dibenzofurans
total heptachlorinated dibenzofurans
total octachlorinated dibenzofurans

2,3,7,8-tetrachlorodibenzo-p-dioxin
1,2,3,7,8-pentachlorodibenzo-p-dioxin
three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin
2,3,7,8-tetrachlorodibenzofuran
2,3,4,7,8-pentachlorodibenzofuran
1,2,3,7,8-pentachlorodibenzofuran
four 2,3,7,8-substituted hexachlorodibenzofurans
1,2,3,4,6,7,8-heptachlorodibenzofuran
1,2,3,4,7,8,9-heptachlorodibenzofuran
1,2,3,4,6,7,8,9-octachlorodibenzofuran

REPORTING FORMAT :

Results are reported in parts per quadrillion (pg/L) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific * and range from 1 ppq to 5 ppq.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the relevant results are reported uncorrected for internal standard recovery.

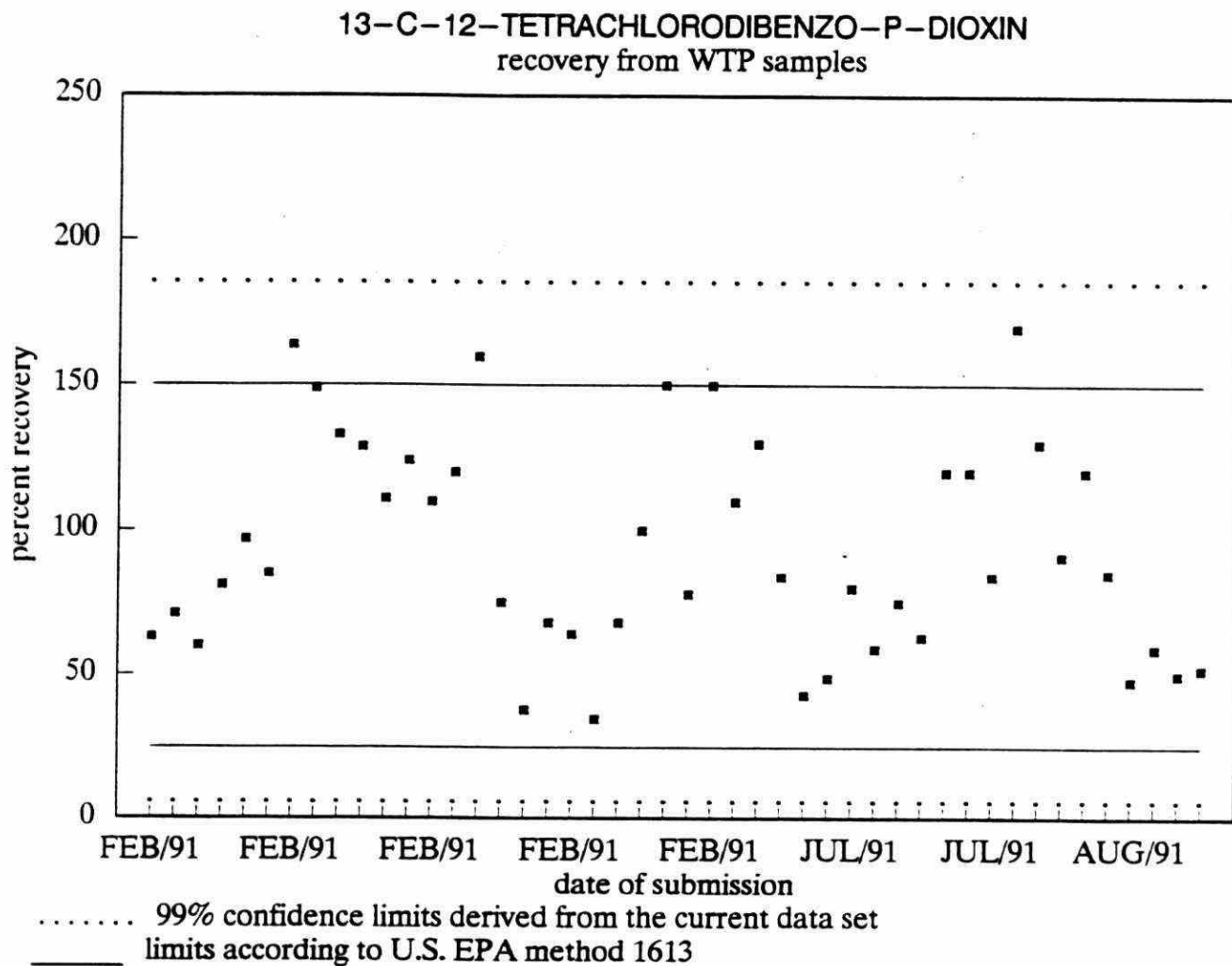
REMARKS : During the period starting January 1991 and ending December 1991, a total of ten method blanks was prepared and tested by the method. For these 10 analyses, no observable responses of any of the target analytes were encountered.

Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts : ¹³C₁₂-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Pentachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Hexachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Heptachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Octachlorodibenzo-p-dioxin (recovery of internal standard)

List of Performance Tables : ¹³C₁₂-2,3,7,8-Tetrachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,7,8-Pentachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin
¹³C₁₂-Octachlorodibenzo-p-dioxin

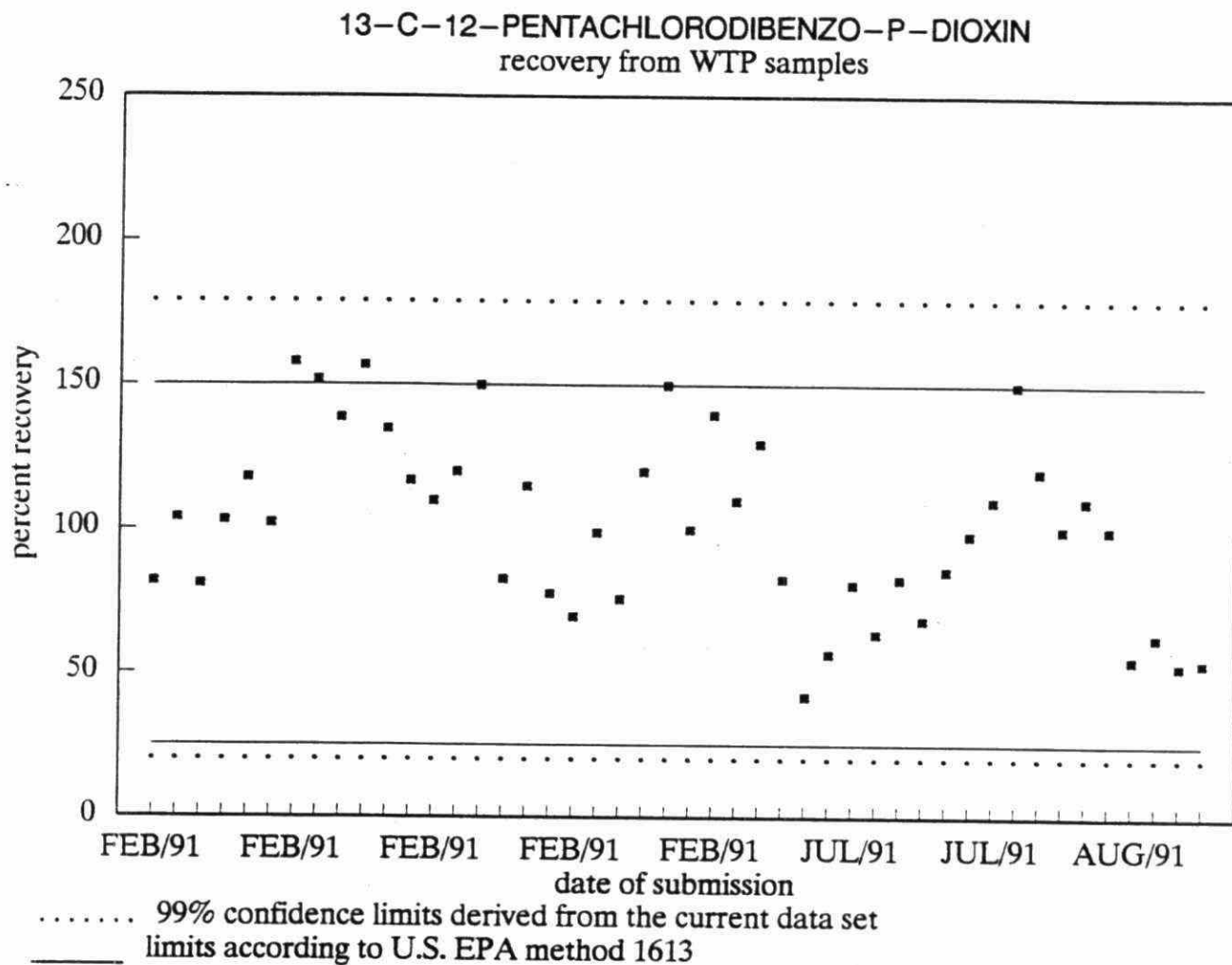
* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -2,3,7,8-tetrachlorodibenzo-p-dioxin
True Concentration	1 ng/L
Number of Observations	117
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	16%
Accuracy (% of expected)	57%

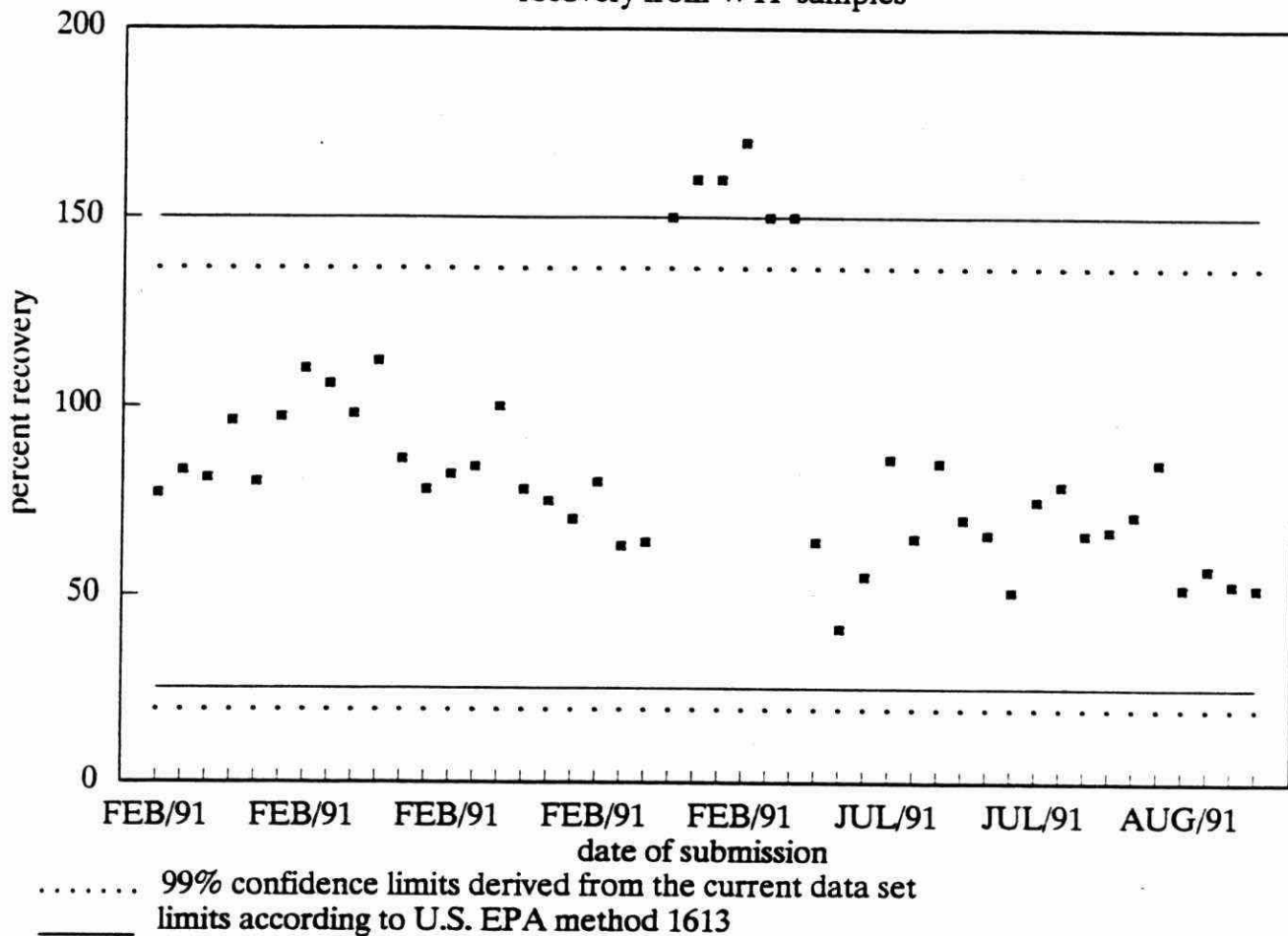


Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,7,8-pentachlorodibenzo-p-dioxin
True Concentration	1 ng/L
Number of Observations	117
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	17%
Accuracy (% of expected)	72%

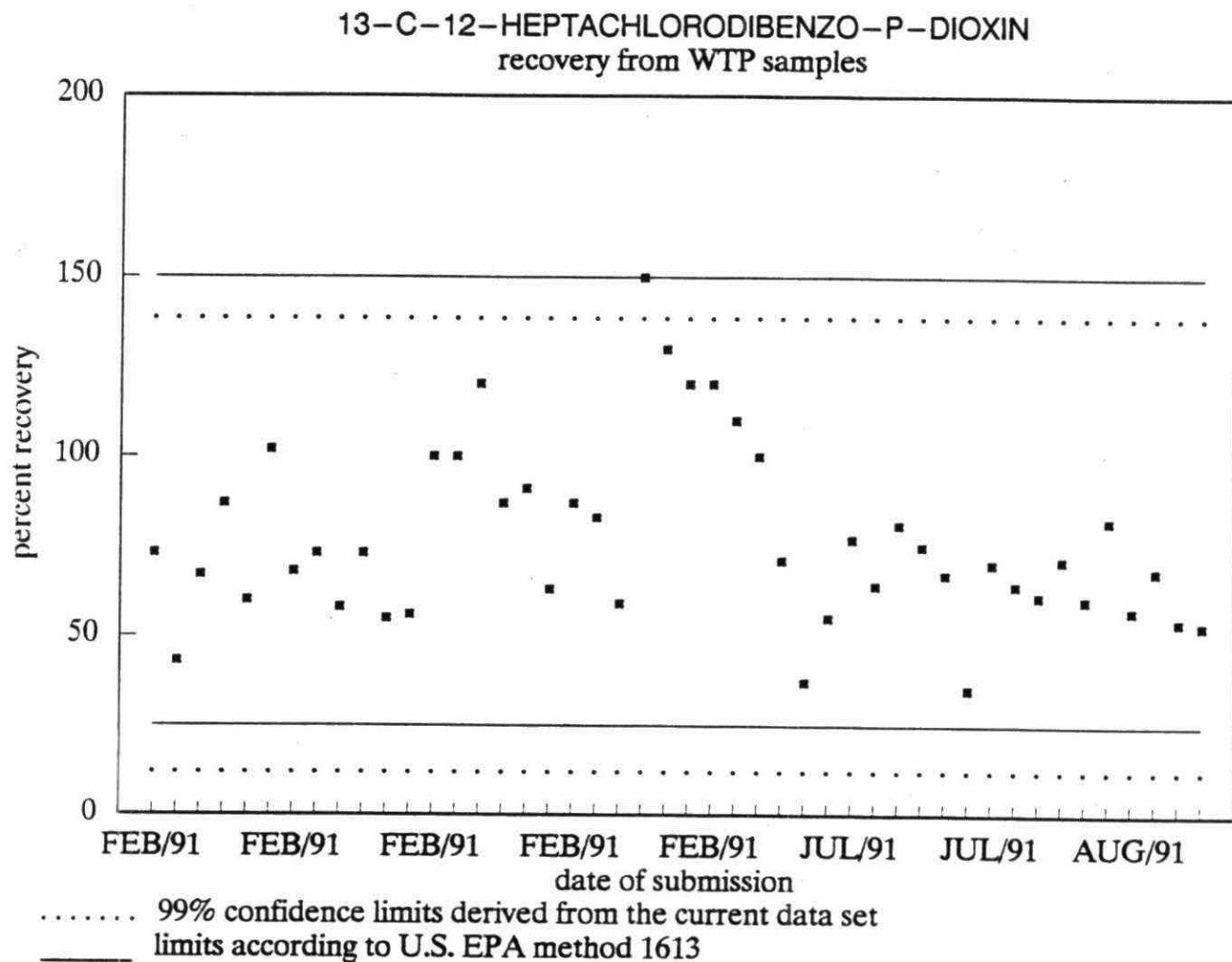
13-C-12-HEXACHLORODIBENZO-P-DIOXIN
recovery from WTP samples



Performance Summary Table

January - December 1991

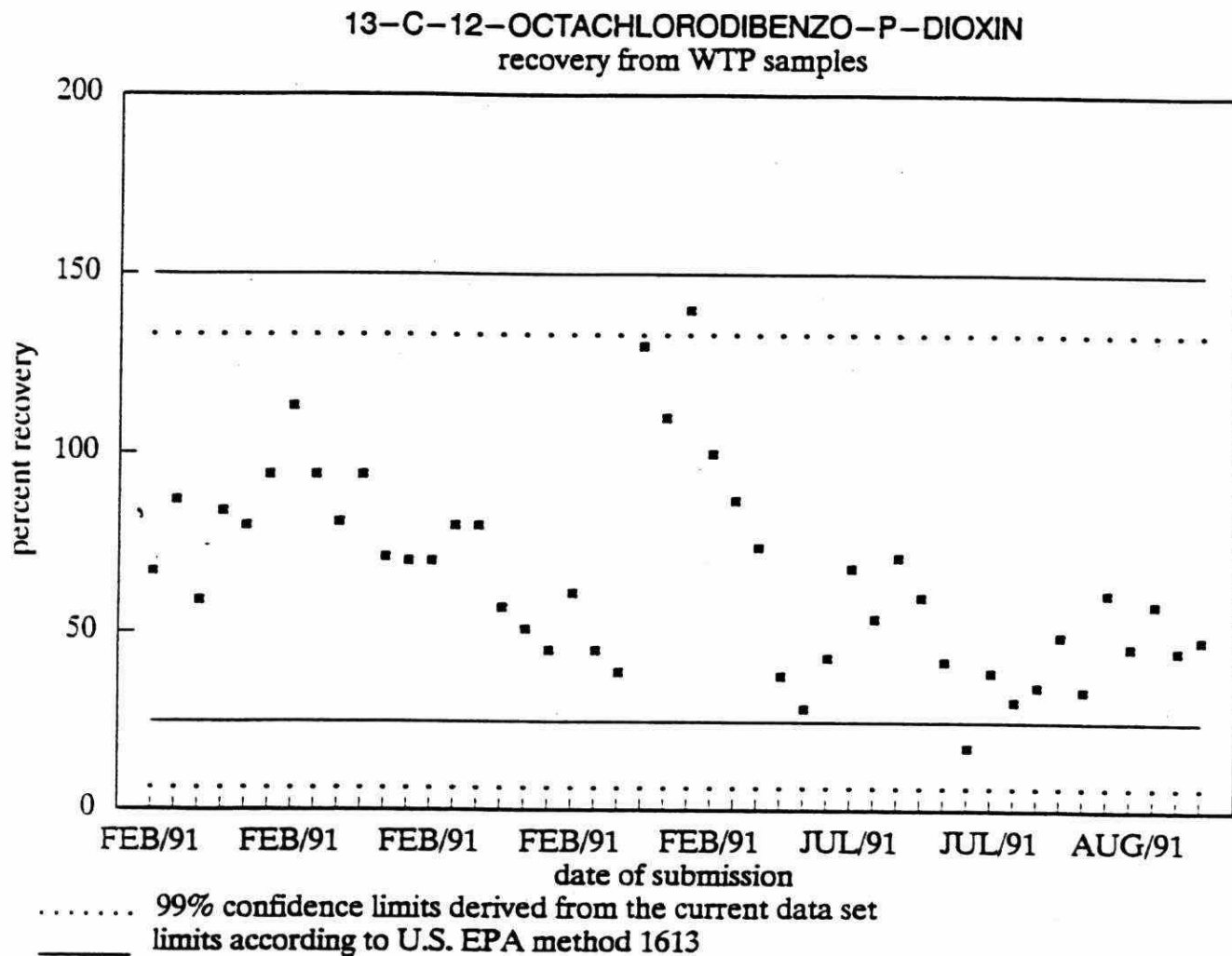
Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin
True Concentration	0.8 ng/L
Number of Observations	117
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	21%
Accuracy (% of expected)	76%



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	$^{13}\text{C}_{12}$ -1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
True Concentration	3 ng/L
Number of Observations	117
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	22%
Accuracy (% of expected)	73%



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -octachlorodibenzo-p-dioxin
True Concentration	2 ng/L
Number of Observations	117
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	21%
Accuracy (% of expected)	69%

METHOD CODE : PWAFFD-E3164A.1

METHOD TITLE: The Determination of Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans in Groundwater and Aqueous Effluent by GC-MS

LABORATORY : Dioxin Unit

SUPERVISOR : E. Reiner

SAMPLE TYPE : groundwater, aqueous industrial or municipal effluent

PRINCIPLE OF THE METHOD :

Sample is filtered to remove visible particulates; the aqueous and particulate portions are processed separately. Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans are extracted from each portion; Soxhlet apparatus is used for filtered particulate portion extraction. Both extracts are dried, concentrated and cleaned up using two-stage chromatographic columns. After clean-up, extracts are evaporated to dryness. The reconstituted extracts are examined by gas chromatography - mass spectrometry.

PARAMETERS MEASURED :

total tetrachlorinated dibenzo-p-dioxins (TCDD)
total pentachlorinated dibenzo-p-dioxins (PCDD)
total hexachlorinated dibenzo-p-dioxins (HxCDD)
total heptachlorinated dibenzo-p-dioxins (HpCDD)
total octachlorinated dibenzo-p-dioxins (OCDD)
total tetrachlorinated dibenzofurans (TCDF)
total pentachlorinated dibenzofurans (PCDF)
total hexachlorinated dibenzofurans (HxCDF)
total heptachlorinated dibenzofurans (HpCDF)
total octachlorinated dibenzofurans (OCDF)

2,3,7,8-tetrachlorodibenzo-p-dioxin
1,2,3,7,8-pentachlorodibenzo-p-dioxin
three 2,3,7,8-substituted hexachlorodibenzo-p-dioxins
1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
1,2,4,6,7,8,9-octachlorodibenzo-p-dioxin
2,3,7,8-tetrachlorodibenzofuran
2,3,4,7,8-pentachlorodibenzofuran
1,2,3,7,8-pentachlorodibenzofuran
four 2,3,7,8-substituted hexachlorodibenzofurans
1,2,3,4,6,7,8-heptachlorodibenzofuran
1,2,3,4,7,8,9-heptachlorodibenzofuran
1,2,3,4,6,7,8,9-octachlorodibenzofuran

REPORTING FORMAT :

Results are reported in parts per quadrillion (pg/L) rounded off to 2 significant figures. The minimum reported levels are sample and analyte specific * and range from 5 ppq to 10 ppq.

QUALITY CONTROL :

The routine quality control operations monitor validity of calibration and consistency in injection volume (injection standard), absence of potential contamination (blanks) and recovery of target analytes (internal standard).

Prior to extraction, each sample is spiked with solution containing isotopically labelled dioxin standards. The recoveries of these isotopically labelled analytes (at least one per each congener group) are monitored. The range for acceptable recoveries is (25-150)%. For the recoveries outside this range, the results are reported not corrected for recovery of internal standard.

REMARKS : Two types of performance limits are displayed on the performance charts. One set was statistically derived from 1991 data set; while the other set is adopted from U.S. EPA method 1613.

List of Performance Charts : ¹³C₁₂-Tetrachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Pentachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Hexachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Heptachlorodibenzo-p-dioxin (recovery of internal standard)
¹³C₁₂-Octachlorodibenzo-p-dioxin (recovery of internal standard)

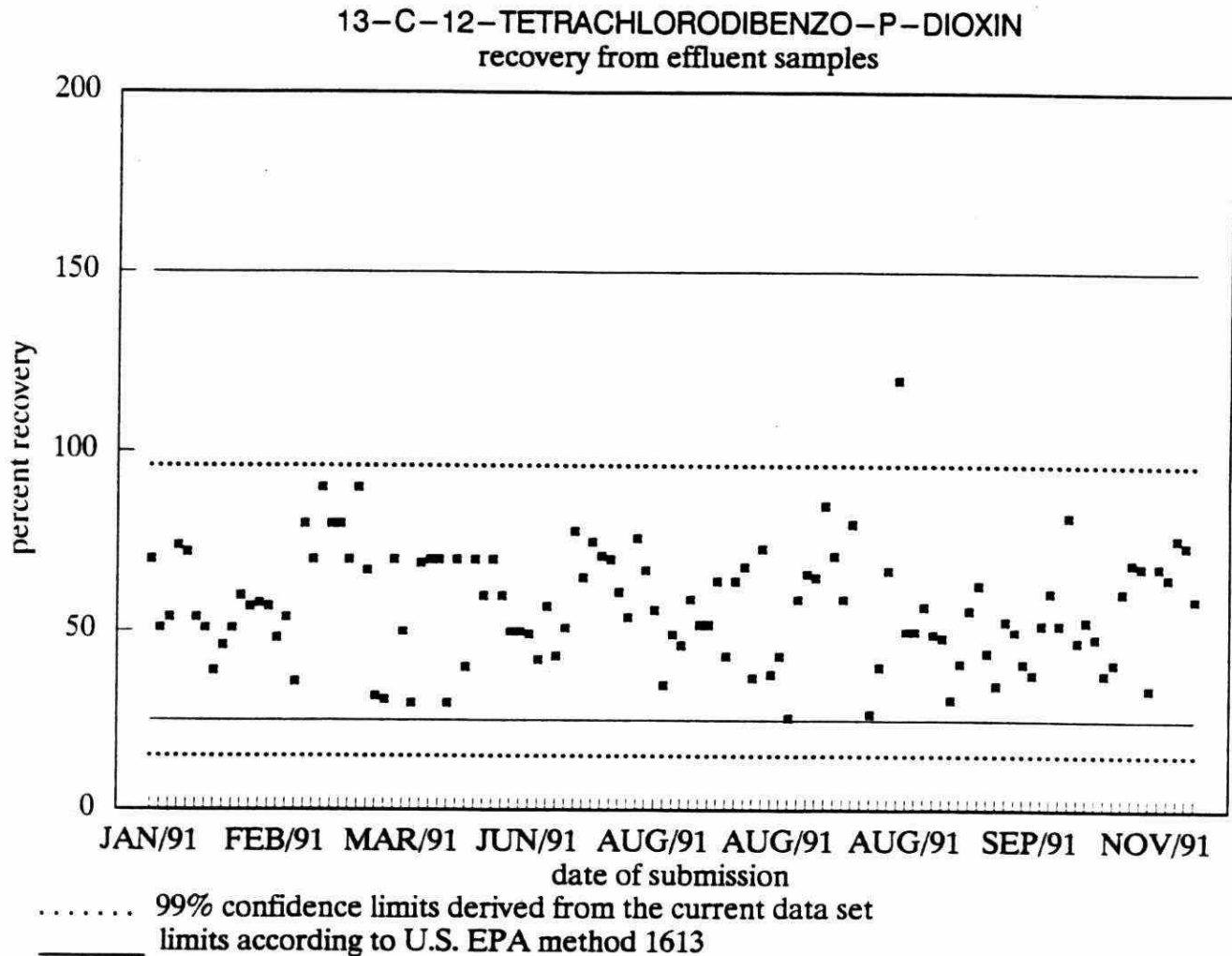
List of Performance Tables : Method Blanks Summary
¹³C₁₂-2,3,7,8-Tetrachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,7,8-Pentachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
¹³C₁₂-1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin
¹³C₁₂-Octachlorodibenzo-p-dioxin

* The minimum reported levels correspond to the amount of analyte that would give most-abundant ion response five times higher than corresponding instrumental noise.

Method Blanks Summary

January 1991 - December 1991

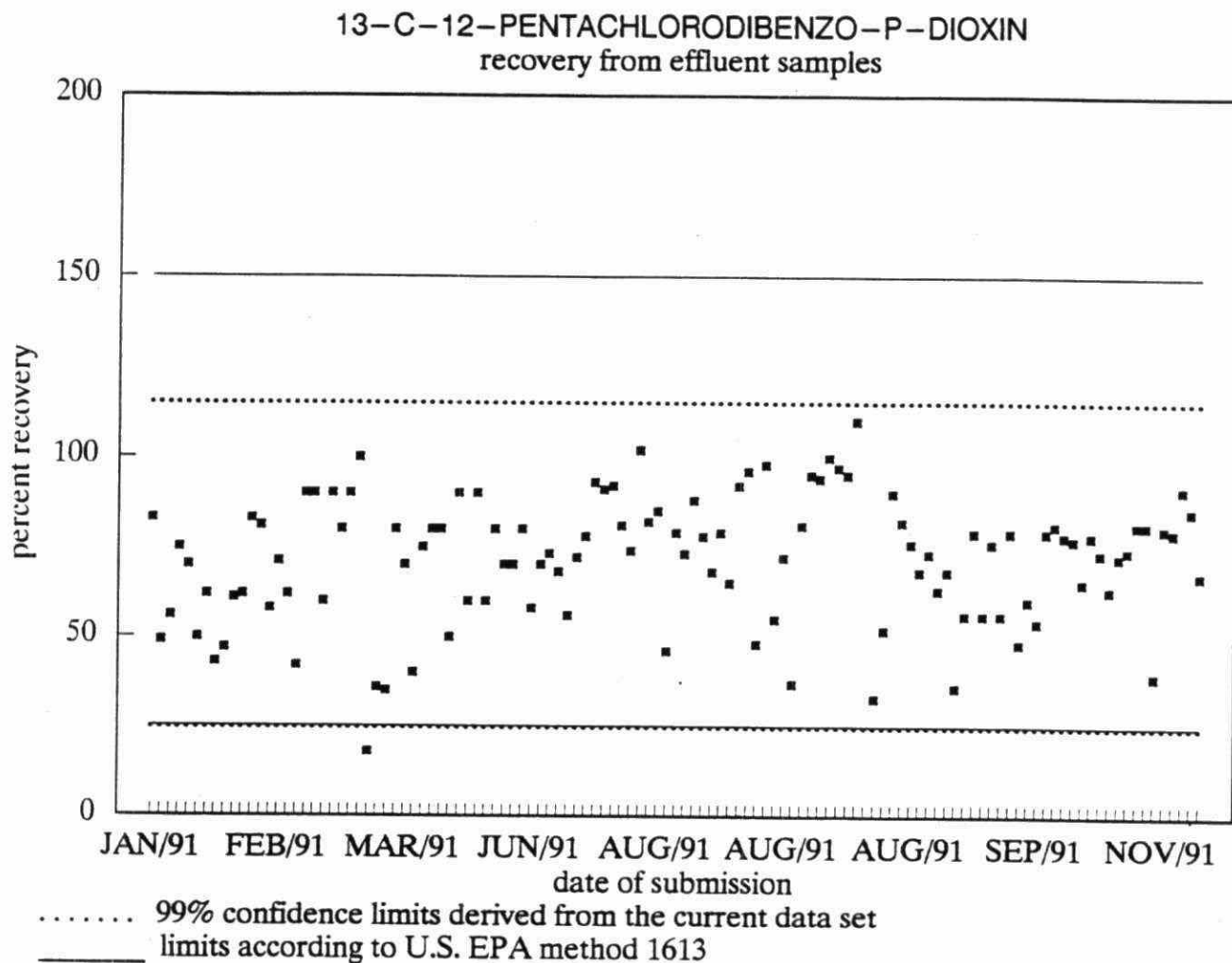
Analyte	Number of Observations	Average Concentration (pg/L)	Standard Deviation (pg/L)
total TCDD	23	0	0
total PCDD	23	0.05	0.21
total HxCDD	23	0.18	0.65
total HpCDD	23	0	0
total OCDD	23	0.18	0.83
total TCDF	23	0	0
total PCDF	23	0	0
total HxCDF	23	0.05	0.21
total HpCDF	23	0.05	0.21
total OCDF	23	0	0



Performance Summary Table

January - December 1991

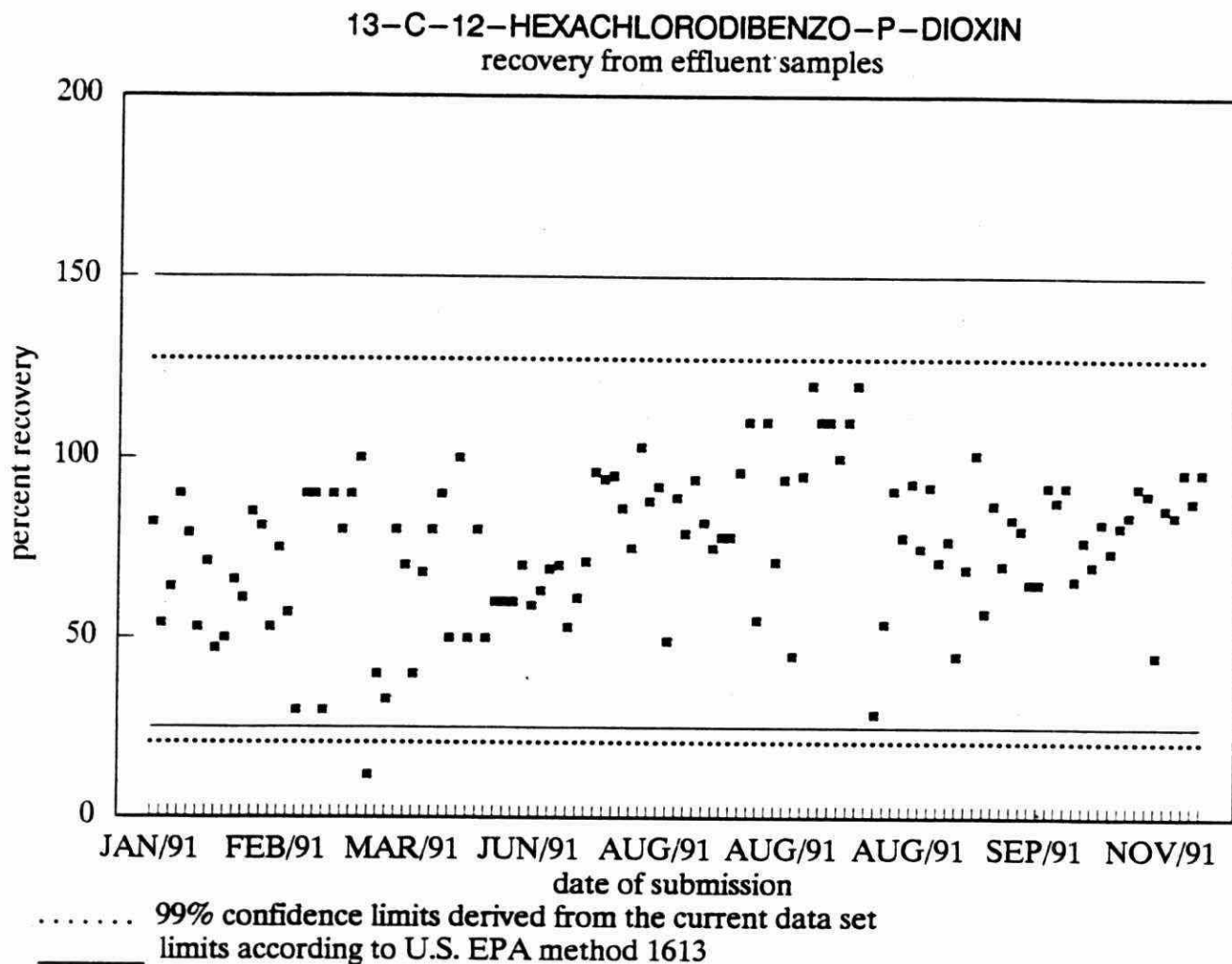
Analyte (Internal Standard)	¹³ C ₁₂ -2,3,7,8-tetrachlorodibenzo-p-dioxin
True Concentration	350 pg/L
Number of Observations	46
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	36%
Accuracy (% of expected)	94%



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,7,8-pentachlorodibenzo-p-dioxin
True Concentration	350 pg/L
Number of Observations	46
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	31%
Accuracy (% of expected)	102%

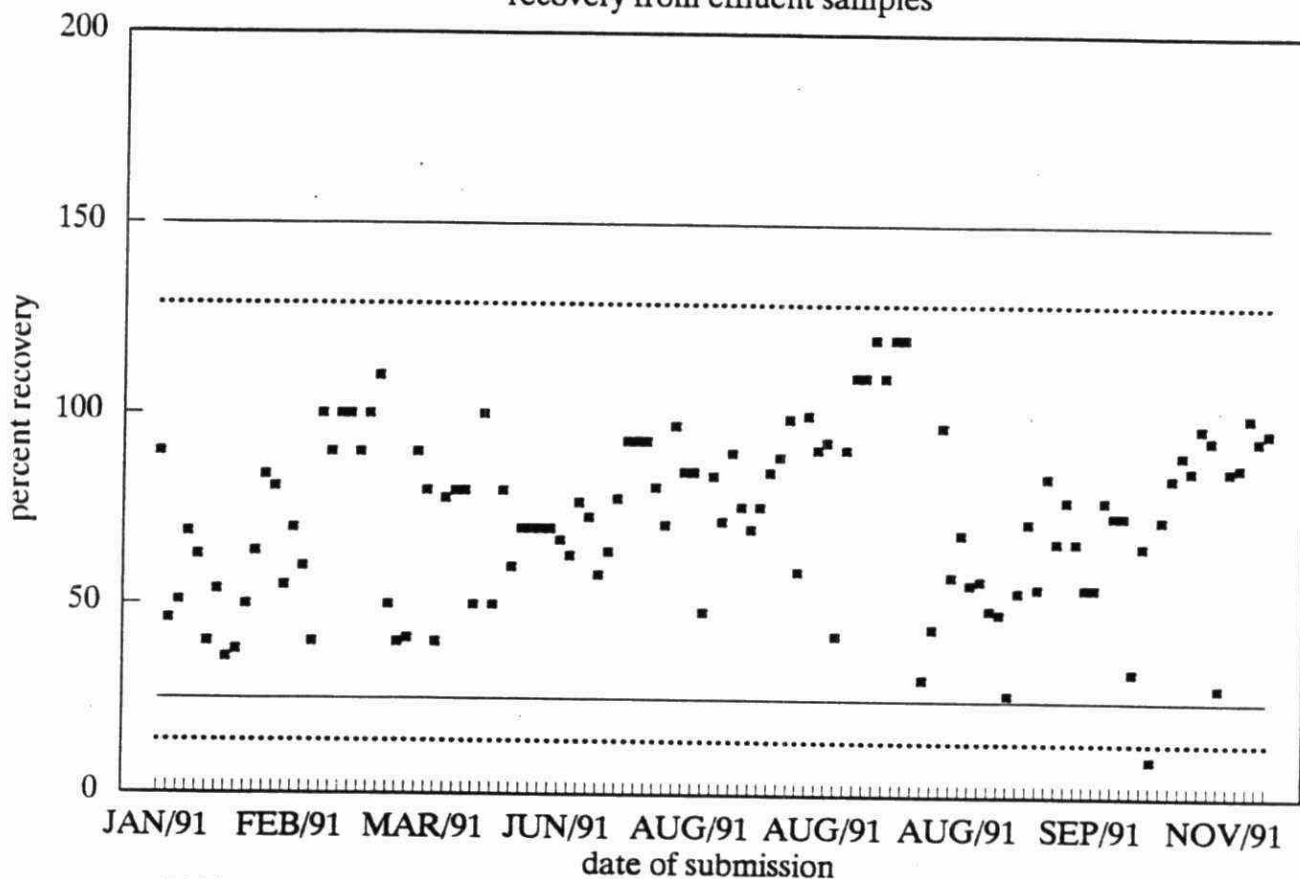


Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -1,2,3,6,7,8-hexachlorodibenzo-p-dioxin
True Concentration	300 pg/L
Number of Observations	46
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	23%
Accuracy (% of expected)	80%

13-C-12-HEPTACHLORODIBENZO-P-DIOXIN
recovery from effluent samples

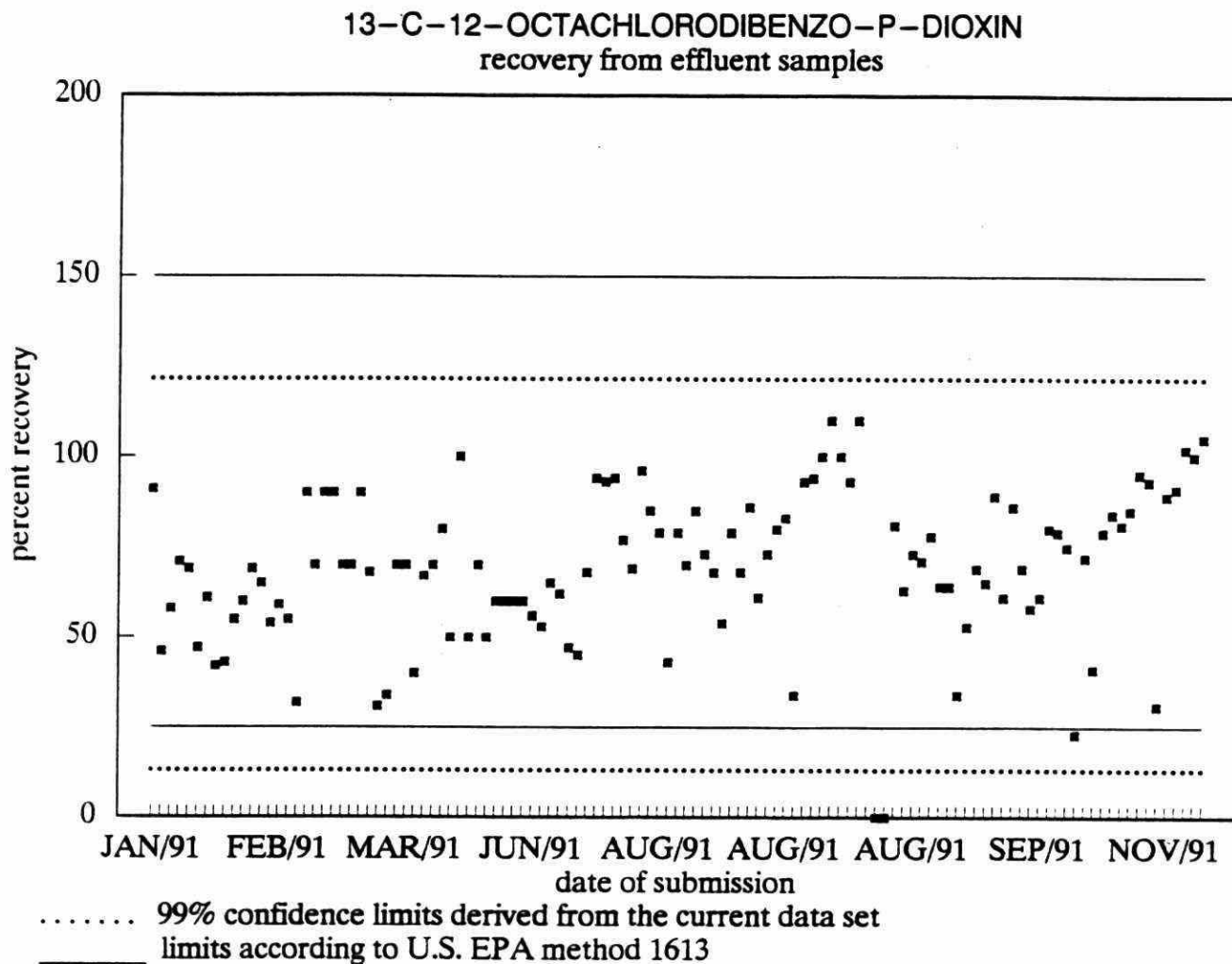


..... 99% confidence limits derived from the current data set
_____ limits according to U.S. EPA method 1613

Performance Summary Table

January - December 1991

Analyte (Internal Standard)	1,2,3,4,6,7,8-heptachlorodibenzo-p-dioxin
True Concentration	1 000 pg/L
Number of Observations	46
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	25%
Accuracy (% of expected)	77%



Performance Summary Table

January - December 1991

Analyte (Internal Standard)	¹³ C ₁₂ -octachlorodibenzo-p-dioxin
True Concentration	600 pg/L
Number of Observations	46
Within-run Rel. Standard Deviation	not available
Between-run Standard Deviation	26%
Accuracy (% of expected)	66%

METHOD CODE : NDMA-E3291A.1

METHOD TITLE: The Determination of N-Nitrosodimethylamine (NDMA) in Drinking Water and in Aqueous Samples by Gas Chromatography / High Resolution Mass Spectrometry (GC/HRMS)

LABORATORY : Mass Spectrometry Unit

SUPERVISOR : V. Taguchi

SAMPLE TYPE : drinking water, aqueous samples

PRINCIPLE OF THE METHOD :

Samples which contain particulate matter are filtered prior to analytical processing.

Sample pH is adjusted to 12 to keep the acidic components in the aqueous phase and the resulting solution is serially extracted with dichloromethane. The dichloromethane extract is washed with a sulphuric acid solution to remove basic components from the organic phase. After being filtered through granular anhydrous sodium sulphate to remove water the extract is concentrated by rotary evaporator and a nitrogen evaporating unit.

The final extract containing the remaining neutral components is analyzed by GC/HRMS. NDMA is quantified by an isotope dilution method.

PARAMETERS MEASURED :

LIS TEST CODE :

MDL (µg/L)

N-Nitrosodimethylamine

MSOBNO (NDMA)

0.005

REPORTING FORMAT :

Results are reported in µg/L rounded off to two significant figures. The lowest reported value is 5×10^{-3} µg/L.

QUALITY CONTROL :

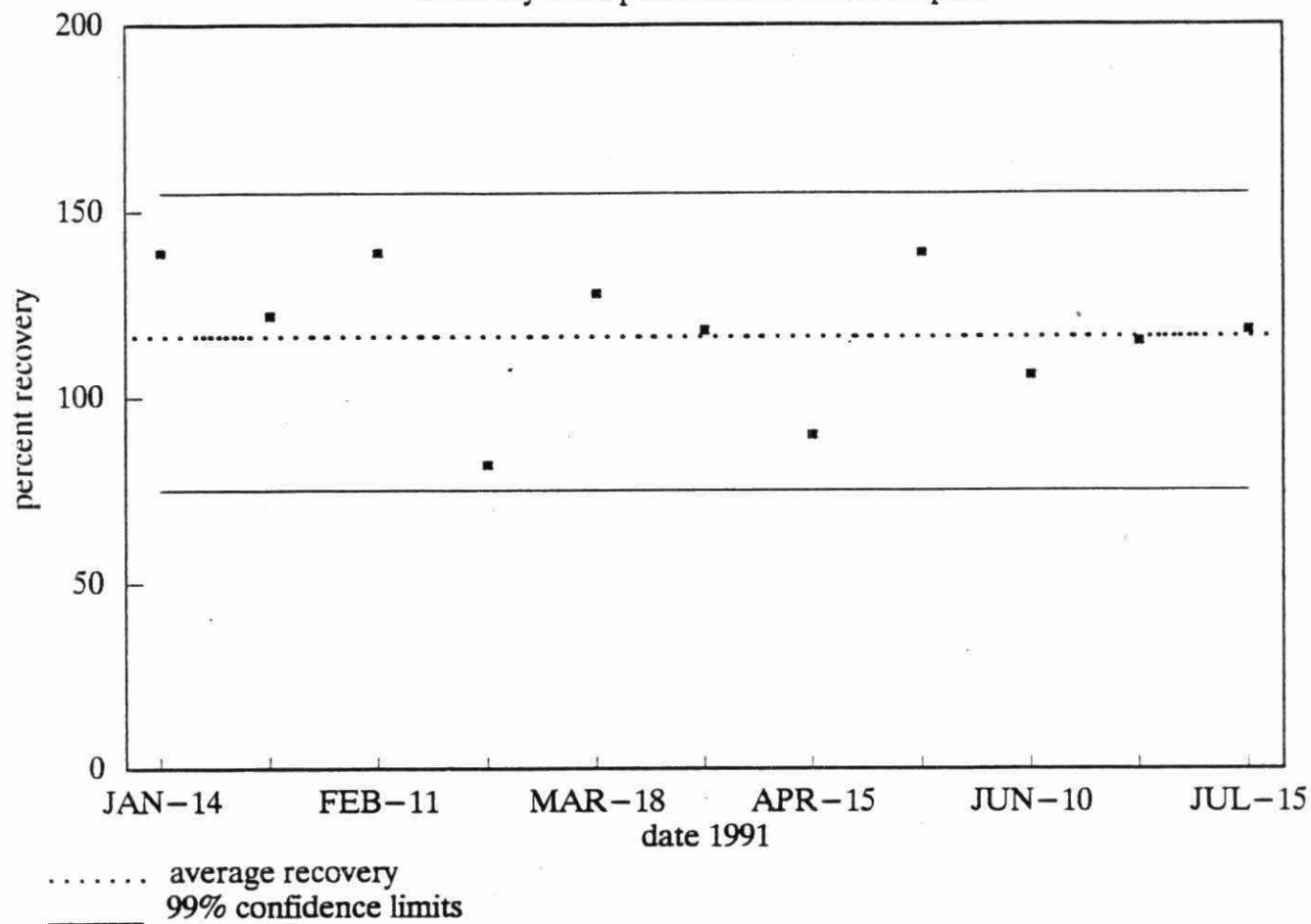
The routine quality control operations monitor validity of calibration (repetitive analysis of calibration standard), size of potential positive bias (method blanks) and maintenance of required instrument sensitivity.

REMARKS : The performance of the method was examined through performance audit samples program and and MOE Interlaboratory Study # 91-2. Both programs were organized by LSB Quality Management Office.

List of Performance Charts : N-Nitrosodimethylamine (recovery from performance audit samples)
N-Nitrosodimethylamine (results for method blanks)

List of Performance Tables : N-Nitrosodimethylamine (intralaboratory and interlaboratory performance
summary)

N-NITROSODIMETHYLAMINE
recovery from performance audit samples



Performance Summary Table

January - December 1991

performance audit samples MSU reproducibility study

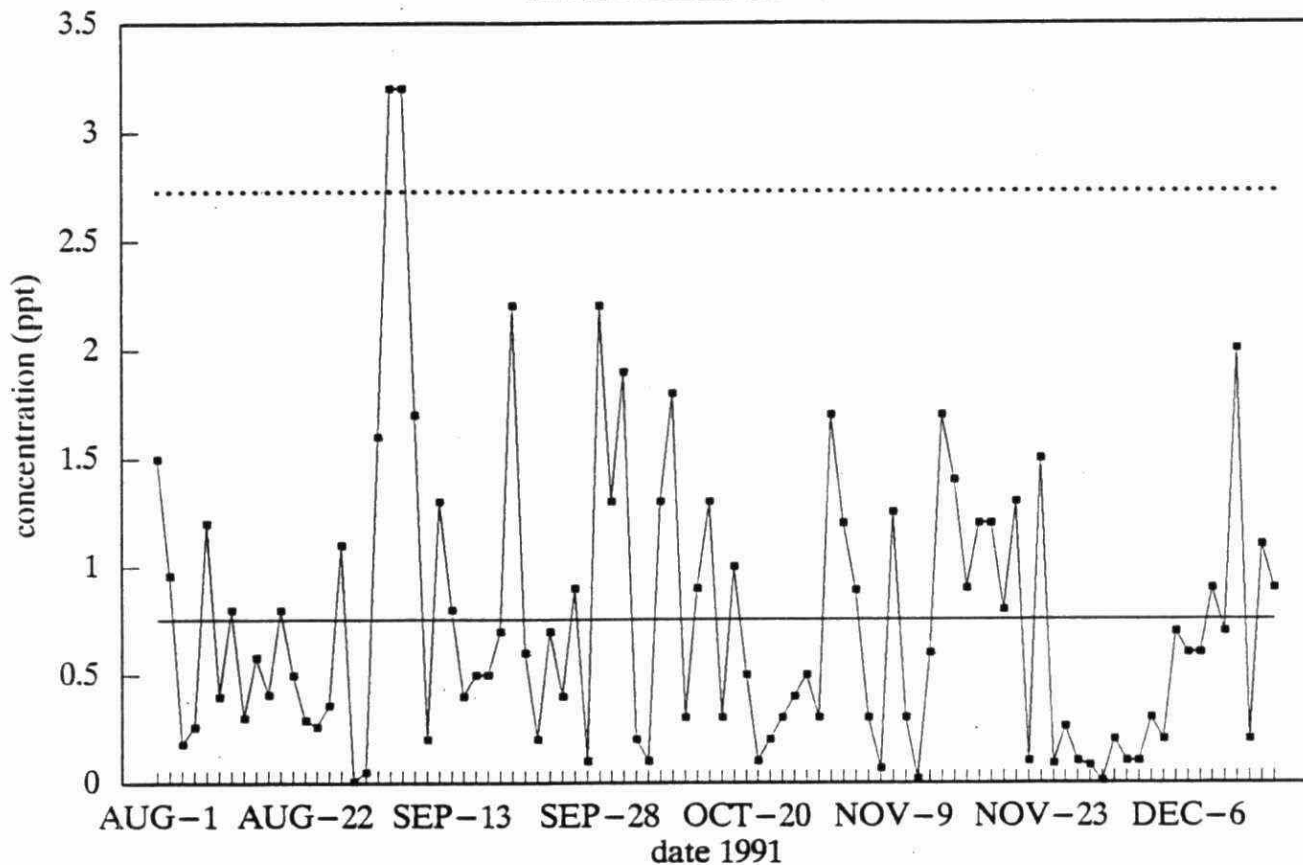
Analyte	N-nitrosodimethylamine	
True Concentration	(0.075 - 0.100) µg/L	69 ^m µg/L
Number of Observations	12	10
Within-run Rel. Standard Deviation	not available	7.5%
Between-run Standard Deviation	18%	19%
Accuracy (% of expected)	118%	156%

Inter-laboratory Comparisons

MOE Inter-laboratory study # 91-2

Number of Participants	13	13
Number of Samples	2	2
Design Value	0.302 µg/L	0.588 µg/L
Inter-laboratory Mean	0.423 µg/L	0.682 µg/L
Inter-laboratory R.S.D.	20%	22%
Intra-laboratory Mean	0.300 µg/L	0.640 µg/L
Intra-laboratory R.S.D.	0%	8%

N-NITROSODIMETHYLAMINE
results from method blanks



———— average concentration found (ppt)
 upper 95%—confidence limit derived from the current data set

Method Blanks Summary

August - December 1991

Analyte	N-nitrosodimethylamine
Number of Observations	92
Mean Concentration	$0.76 \times 10^{-3} \mu\text{g/L}$
Standard Deviation	$0.67 \times 10^{-3} \mu\text{g/L}$

METHOD CODE : SMY-E3186A.1
METHOD TITLE: The Determination of Extractable Organics in Drinking Water, Aqueous Samples, Soil and Sediment by Gas Chromatography / Mass Spectrometry (GC/MS)
LABORATORY : Mass Spectrometry Unit
SUPERVISOR : V. Taguchi
SAMPLE TYPE : drinking water, aqueous samples, soil, sediment

PRINCIPLE OF THE METHOD :

The method involves extraction of the organic components from their respective matrices with an organic solvent and analysis of the concentrated extract by gas chromatography / full scan mass spectrometry.

Drinking water and aqueous samples are initially adjusted to pH 12. The base/neutral components are serially extracted with dichloromethane. The remaining aqueous fraction is then adjusted to pH 2 and the acidic components are serially extracted with dichloromethane. The extracts are filtered through anhydrous sodium sulphate to remove water and then concentrated by rotary evaporator and a nitrogen evaporating unit. The final extracts are analyzed by GC/FS-MS.

Soil and sediment samples are extracted with toluene in a Soxhlet/Dean-Stark apparatus. The concentrated extracts are analyzed by GC/FS-MS.

The extractable organics are reported according to their compound names and compound classes. The concentrations of the components are approximate and are calculated relative to the internal standard.

PARAMETERS MEASURED :

extractable organics analyzable by GC/MS

LIS TEST CODE :

PBEXT

REPORTING FORMAT :

The extractable organics are reported according to their compound names, CAS registry numbers and compound classes*. The concentrations of all components are approximate and are reported in $\mu\text{g/L}$ (resp. ng/g for soil and sediment samples) to one significant figure with the prefix "A-". The minimum reported concentrations (calculated relatively to the internal standard) depend on sample matrix and range from $0.1 \mu\text{g/L}$ to $1 \mu\text{g/L}$.

QUALITY CONTROL :

The routine quality control operations monitor presence of potential interferences in the method blanks, relative extraction efficiencies (surrogates), instrumental performance (MS tuning characteristics / autotune /, reference standard solution analysis).

The ratio of d_6 -NDMA to the internal standard d_{10} -phenanthrene is used to monitor extraction efficiencies (recoveries) of a polar compound relative to a non-polar compound and is also an indicator for chromatographic performance.

REMARKS : The performance of the method was periodically examined through performance audit samples program administered by LSB Quality Management Office.

List of Performance Tables : Performance Audit Samples Summary

Clement, R.E.; Taguchi, V.Y.; Techniques for the Gas Chromatography - Mass Spectrometry Identification of Organic Compounds in Effluents; Environment Ontario, July 1989

Performance Audit Samples Summary, January - December 1991

analyte	number of tests	concentration range (µg/L)	analyte identified [percent of tests]	recovery within (10-200)% (% of identified)
bis(2-chloroethyl) ether	20	3.0 - 6.0	20 [100%]	100 %
1,3-dichlorobenzene	20	3.0 - 6.0	20 [100%]	100 %
1,2-dichlorobenzene	20	3.0 - 6.0	20 [100%]	100 %
N-nitroso-di-n-propylamine	20	3.0 - 6.0	19 [95%]	95 %
isophorone	20	3.0 - 6.0	16 [80%]	100 %
bis(2-chloroethoxy)methane	20	3.0 - 6.0	20 [100%]	100 %
1,2,4-trichlorobenzene	20	3.0 - 6.0	20 [100%]	95 %
hexachlorobutadiene	20	3.0 - 6.0	18 [90%]	100 %
2-chloronaphthalene	20	3.0 - 6.0	18 [90%]	100 %
2,6-dinitrotoluene	20	3.0 - 6.0	18 [90%]	100 %
2,4-dinitrotoluene	20	3.0 - 6.0	20 [100%]	100 %
diethyl phthalate	20	3.0 - 6.0	20 [100%]	100 %
hexachlorobenzene	20	3.0 - 6.0	20 [100%]	100 %
phenanthrene	20	3.0 - 6.0	20 [100%]	100 %
dibutyl phthalate	20	3.0 - 6.0	17 [85%]	100 %
pyrene	20	3.0 - 6.0	19 [95%]	100 %
benzo(a)anthracene	18	3.0 - 6.0	15 [83%]	100 %
dioctyl phthalate	20	3.0 - 6.0	16 [80%]	89 %
benzo(k)fluoranthene	20	3.0 - 6.0	16 [80%]	89 %
base/neutral extractables total	378		352 [93%]	98 %
phenol	20	1.9 - 4.0	19 [95%]	90 %
2-chlorophenol	20	2.5 - 5.5	20 [100%]	90 %
2,4,6-trichlorophenol	20	1.5 - 3.2	20 [100%]	95 %
p-chloro-m-cresol	20	2.5 - 5.0	19 [95%]	95 %
2-nitrophenol	20	2.0 - 4.0	15 [75%]	100 %
2,4-dichlorophenol	20	2.3 - 4.7	19 [95%]	100 %
4-nitrophenol	20	0.5 - 1.0	4 [20%]	75 %
2,4-dimethylphenol	20	3.5 - 7.7	14 [70%]	93 %
pentachlorophenol	20	3.0 - 6.0	13 [65%]	92 %
acidic extractables total	180		143 [79%]	94 %

METHOD CODE : SMY-E3189A.1
METHOD TITLE: The Determination of Volatile Organics in Drinking Water and Aqueous Samples by Purge-and-Trap Gas Chromatography / Mass Spectrometry (P&T/GC/MS)
LABORATORY : Mass Spectrometry Unit
SUPERVISOR : V. Taguchi
SAMPLE TYPE : drinking water, aqueous samples

PRINCIPLE OF THE METHOD :

The volatile organics are purged from the matrix with helium gas, isolated onto multi-layered trap(s) and then thermally desorbed and analyzed by gas chromatography / full scan mass spectrometry.

The volatile organics are reported according to their compound names and compound classes. The concentrations of the components are approximate and are calculated relative to the internal standard.

PARAMETERS MEASURED :

volatile organics analyzable by P&T/GC/MS

LIS TEST CODE :

PBVOL

REPORTING FORMAT :

The volatile organics are reported according to their compound names, CAS registry numbers and compound classes *. The concentrations of all components are approximate and are reported to one significant figure with the prefix "A-". The minimum reported concentrations (calculated relatively to the internal standard) depend on sample matrix and range from 0.1 µg/L to 1 µg/L.

QUALITY CONTROL :

The routine quality control operations monitor the presence of potential interferences (blanks), relative purging efficiencies (surrogates), instrumental performance (MS tuning characteristics / autotune /, reference standard solution analysis).

* Clement, R.E.; Taguchi, V.Y.; Techniques for the Gas Chromatography - Mass Spectrometry Identification of Organic Compounds in Effluents; Environment Ontario, July 1989

The ratio of d_4 -1,2-dichloroethane to the internal standard is used to monitor relative purging efficiencies (recoveries) and is also an indicator for the relative response factors of the components. For MISA samples, 1,3-dichlorobutane is used as an internal standard, for all other samples, the internal standard is d_{10} -ethylbenzene.

REMARKS : The performance of the method was periodically examined through performance audit samples program administered by LSB Quality Management Office.

List of Performance Tables : Performance Audit Samples Summary

Performance Audit Samples Summary, January - December 1991

analyte	number of tests	concentration range (µg/L)	analyte identified [percent of tests]	recovery within (50-150)% (% of identified)
1,2-dichloroethane	9	0.9 - 2.0	9 [100%]	100 %
chloroform	9	6.5 - 15.0	9 [100%]	89 %
1,1,1-trichloroethane	9	0.6 - 1.4	9 [100%]	89 %
trichloroethylene	9	0.4 - 1.0	9 [100%]	100 %
carbon tetrachloride	9	1.3 - 3.0	9 [100%]	100 %
tetrachloroethylene	9	0.8 - 1.7	9 [100%]	100 %
bromodichloromethane	9	0.4 - 1.0	9 [100%]	100 %
dibromochloromethane	9	1.0 - 2.4	9 [100%]	100 %
bromoform	9	1.5 - 3.3	9 [100%]	100 %
o-xylene	9	0.9 - 2.0	9 [100%]	100 %
m&p-xylene	9	1.7 - 3.7	9 [100%]	100 %
1,2-dichlorobenzene	9	0.9 - 2.1	9 [100%]	100 %
1,3-dichlorobenzene	9	0.9 - 2.0	9 [100%]	100 %
1,4-dichlorobenzene	9	0.9 - 2.0	9 [100%]	100 %
1,2-dibromoethane	9	0.9 - 2.0	9 [100%]	100 %
volatile organics total	135		135 [100%]	98.5 %

GLOSSARY OF TERMS

accuracy	proximity to the true value expressed as average percent recovery or average percent of expected
average (mean)	sum of the measurements divided by the number of measurements
between-run experiment	samples are prepared by different technicians, and the instrumental analyses take place under different calibrations of the analytical system
between-run r.s.d.	measure of reproducibility of a method
calibration solution	solution containing target analyte(s) for a particular method at concentration(s) that will produce response(s) falling within the linear range of the instrument. This solution is used to calibrate the instrument response with respect to the analyte concentration.
calibration check solution	solution which has composition similar to the calibration solution and which is prepared independently of calibration solution. It is used to check performance of the instrument, especially the validity of current calibration.
fortified method blank	synthetic sample prepared by adding known quantities of target analytes of the method to the interference-free matrix
internal standard	known amount of a compound, that is assumed to have identical chemical and physical properties with the analyte(s) of interest, is added to the sample prior to sample processing. The recovery of this compound from the sample is used for correction of the final results.
MDL	method detection limit. MDL marks the concentration level above which one can conclude that a measured result indicates the presence of analyte in the sample with a specified confidence (99%).
method code	Analytical Methods Catalogue Code used within Ontario Ministry of the Environment
percent recovery	ratio of the concentration obtained by the experiment to the theoretical concentration, multiplied by one hundred
performance charts	graphical presentation of the individual results of the analyses of fortified method blanks or internal standards. The x-axis on the chart represents the date, the y-axis outlines percent recovery. The average and 99% confidence limits are displayed as well.

relative standard deviation	measure of spread of a population. The square root of the squared sum of the measurements minus the sum of squared measurements, divided by the number of measurements minus one.
T value	level below which analytical results represent trace values; additional data are needed for valid interpretation (see Code of Practice for Environmental Laboratories, September 1989, Ontario Ministry of the Environment)
upper and lower 99% confidence limit, UL (LL)	$UL (LL) = X + (-) t \times s$ X,s represent the average and the standard deviation of the replicate measurement; $t_{(n-1, \alpha=0.01)}$ is the Student's t-value appropriate for a 99% confidence level and the given number of degrees of freedom <u>n</u>
within-run experiment	samples are prepared and analyzed by a single technician, and the instrumental analyses take place within one calibration of the analytical system
within-run r. s. d.	measure of repeatability of a method
W value	minimum reported level (see Code of Practice for Environmental Laboratories, September 1989, Ontario Ministry of the Environment)

HAZARDOUS CONTAMINANTS
COORDINATION BRANCH
135 ST. CLAIR AVENUE WEST
TORONTO, ONTARIO M4V 1P5

TD
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